

Twin Screw Extruder Production of MTTP Decoy Flares SERDP WP-1240

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FINAL REPORT

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ACRONYMS AND ABBREVIATIONS

BBP	benzylbutylphthalate
DMP	dimethylphthalate
DOA	dioctyladipate
DTA	Differential Thermal Analyses
ECD	electron capture detector
EPA	Environmental Protection Agency
ESD	electro-static discharge
ESTCP	Environmental Security Technology Certification Program
FF	first fire
GAP	
HAP	Hazardous Air Pollutant
IC	intermediate charge
IR	infrared
LIW	loss in weight
MTH	Magnesium-Teflon-Hytemp
MTTP	Magnesium-Teflon-Thermoplastic
NSWC	Naval Surface Warfare Center
P2	Pollution Prevention
PCB	polychlorinated biphenyl
PPCB	Plant Process Control Board
PTFE	polytetrafluoroethylene
PVAc	polyvinyl acetate
PVC	polyvinyl chloride
R&D	Research and Development
SAC	Safety Advisory Committee
TP	thermoplastic
TSE	twin-screw extruder
VOC	volatile organic compound

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1.0 EXECUTIVE SUMMARY

A formulation and process have been developed to demonstrate the feasibility of producing an aircraft decoy composition via a solvent-free twin-screw extrusion process, significantly improving the environmental and safety hazards associated with the standard batch process currently used for production. The formulation employs magnesium, Teflon[®], and a thermoplastic (TP) binder, hence the designation Magnesium-Teflon-Thermoplastic (MTTP). After screening a number of candidate TPs, the TP selected was polyvinyl chloride (PVC)-copolyvinyl acetate (PVAc). A combustion residue analysis was conducted on the MTTP formulation via Environmental Protection Agency (EPA) Method 8280 and Method 8080 to determine if dioxins or furans were produced; none were observed down to a detection limit of 0.1 ppm. The composition was produced during multiple runs on a 19-mm twin-screw extruder (TSE). Burn time and radiant energy were measured on a number of extruder runs. While the burn times were longer for the MTTP composition than the baseline MTV composition, the radiant energy output (J/sr-g) was comparable. An interim hazard classification was obtained. Eight pounds of material were packaged and shipped to ARDEC; slightly over eleven pounds of material were packaged and shipped to Naval Surface Warfare Center (NSWC) Crane for subscale characterization and evaluation in their respective flare configurations. NSWC Crane completed sensitivity and Differential Thermal Analyses (DTA) testing and found the MTTP composition to be similar to the baseline infrared (IR) Flare Composition 757JC. ARDEC completed static and wind tunnel testing of 32 full-size MTTP flares and showed the MTTP IR output slightly outperformed the standard Magnesium-Teflon-Hytemp (MTH) flare under static and dynamic test environments. The burn time was average, and the MTTP visible light output was 93% of the MTH flare. ARDEC found the performance of MTTP satisfactory in the full-size flare configuration.

1.1 PROJECT BACKGROUND

Aircraft decoy flares whose IR emissions derive largely from reactions between magnesium, Teflon[®], and Viton[®] or Hytemp[®] binder (MTV or MTH) continue to be important countermeasures to protect military helicopters and fixed-wing aircraft against heat-seeking missiles. Environmental and safety concerns are major drawbacks to the current solvent-based processing technology for manufacturing these compositions. This is due primarily to use of large quantities of acetone and hexane, a Hazardous Air Pollutant (HAP), in the manufacturing process. Significant amounts of these flammable solvents vaporize into the atmosphere, where they pose environmental, personnel health, and safety hazards.

The production of MTV/MTH flares has resulted in numerous events involving personnel injury and death. Incidents arising from accidental ignition of solvent vapors continue to be the bane of manufacturers of MTV decoy flares. Current manufacturing processes, although improved over historical methods, are batch processes that require transfers of large quantities of highly flammable solvents from one container to another. Risks of accidental ignitions are high, but eliminating these risks has been shown to be both difficult and expensive.

1.2 OBJECTIVE

The objective of this effort is to develop an environmentally acceptable decoy flare formulation and process to produce aircraft decoy flares without the use of HAP or Volatile Organic Compounds (VOC). A continuous TSE will be used to compound magnesium, Teflon[®], and a TP binder into formulations (MTTP) for decoy flares that meet current MTV or MTH countermeasure product specifications. The process will significantly reduce the air pollution, personnel health hazard, loss of life through solvent fires, and hazardous waste production associated with MTV or MTH production.

During the first phase of this program, a suitable formulation is to be identified as well as a solvent-free process involving the TSE to produce it. The formulation is to be produced and shipped to ARDEC and NSWC Crane in the second phase. In the final phase of the program, ARDEC and NSWC Crane will press the material into flare grains and test their performance relative fielded decoys.

1.3 TECHNICAL APPROACH

Thermoplastic binders have the advantage that they can be processed in the TSE without the use of solvents. Elimination of HAPs and VOCs from the process will meet the environmental goal of this program. The methodology to develop an environmentally acceptable formulation and process is described as follows:

i. Formulation Development - MTTP decoy flare compositions will be formulated with magnesium powder, polytetrafluoroethylene (Teflon[®]), and TP binders in accordance with the percentages studied under previous Thiokol IR&D programs. In accordance with a preliminary environmental assessment, each MTTP flare ingredient has low toxicity and presents no long-term health hazards. Potential TP candidate types including polystyrene-based resins and ethylene-vinyl acetate copolymers will be investigated. These TPs and others to be investigated are widely used in commercial products and have high carbon content that is desirable for decoy flare gray-body emission. Magnesium and Teflon[®] powders that are in conformance to the military specifications or drawings will be used. The physical and chemical properties (particle size, density, melt point, soft point, decomposition temperature, etc.) and safety characterization (electrostatic discharge, friction, impact, and simulated bulk auto-ignition) of each ingredient and representative MTTP formulations will be acquired in addition to performance data (burn time, radiant intensity, radiant energy, etc) to identify top candidates to be investigated further for ease of and safety during processing.

ii. Pre-extrusion Computer and Rheology Modeling

(1) Torque and Capillary Rheometer Modeling - Torque and capillary rheometry of potential MTTP compositions along with burn performance described above will be used to downselect an MTTP formulation to be advanced to the TSE. A thorough understanding of the material's physical properties is needed as input for the extruder analytical model.

The material's rheology will be initially characterized using a parallel plate (torque) rheometer. The torque rheometer compounds materials in a very similar fashion to the TSE and is extremely valuable in obtaining relevant rheometric data directly applicable to the TSE.

The capillary rheometer consists of a ram extruder that forces the pyrotechnic emulsion through a series of capillary dies with different lengths (L) and diameters (D). In these material selection runs, the pressure will be set and the resulting flow rate will be measured. These results tell us how the material will behave in the extruder and will be used in computer modeling.

(2) Akro-Co-Twin Screw Computer Modeling - This program models complex fluid flow in a dynamic environment specifically for co-rotating TSEs. It can model the temperature, fill factor, pressure gradient, melt, and residence profile along the longitudinal axis of the screw. The program contains an internal database consisting of a wide range of makes and models of extruders. Different barrel and screw element configurations can be constructed for each extruder model. Required inputs for the program are material rheology, thermodynamic properties, and operation conditions of the extruder such as screw speed, flow rate, barrel temperature, and die pressure.

iii. TSE Process Development –Initial studies will be made to characterize the accuracy of feeding individual raw materials. Feeder performance will be optimized through evaluation of a variety of hardware and software configurations for each feed stream. Once the feed streams have been defined and the feed systems optimized, compounding and extrusion efforts will proceed using the Thiokol 19-mm TSE. The order of adding ingredients to the extruder will be investigated during the study. The combination of the forward, reverse, and conveying screws will also be studied along with the barrel temperature to established optimal extrusion configuration. Extruded material will be collected in the product collection tunnel for granulation.

iv. MTTP Scrap Recycling - Energetic scraps have always been produced by conventional batch processes. They cannot be reprocessed and must be disposed of in licensed open burning grounds. The thermal characteristics of MTTP compositions will not be changed by the proposed process and thus can be recycled. The reprocessed and virgin MTTP granules will be characterized for comparison in performance (burn time, rise time, spectral output), physical (density, compression strength), and thermal characteristics (DTA, TGA thermograms).

v. Combustion Residue – The MTTP combustion residue will be analyzed according to EPA methods 8280 and 8080 to determine if dioxins or furans are produced in burning the new composition.

vi. Flare Characterization - The representative flare composition will be shipped to Army and Navy test facilities for characterization and comparison to standard MTV or MTH flares. In addition to Thiokol standard laboratory safety tests, the Government will conduct limited safety tests on the samples to verify their safety characteristics prior to processing in the

ARDEC/Crane pyrotechnic processing and loading plants. Characterization of extruded MTTP compositions will include functionality and grain integrity, as described below.

(1) Static and Dynamic Radiometric Performance (ARDEC/NSWC Crane) - Extruded MTTP granules will be consolidated into fully configured Navy and Army flare pellets for testing. Static radiometric outputs including burn time, rise time, and corresponding IR intensity will be collected for each composition in accordance with military specifications. Both temperature conditioned and unconditioned samples will be tested. The control samples are made from the existing MTH or MTV compositions.

(2) Mechanical Compression Strength (ARDEC) - Extruded MTTP granules will be consolidated at ~11,000 psi to 3/8" by 3/8" pellets for testing in an Instron Mechanical Property System. A pressed pellet will be placed on a sample platform of the system and compressed by a slowly released overhead load cell until it is deformed or crushed. The load at this point is the crush strength (compression strength) of pellet. Five pellets for each composition will be tested. The control samples are made from the existing MTH or MTV compositions.

The conclusion of the MTTP flare characterization by the Army and Navy will represent the completion of the program.

1.4 PROJECT ACCOMPLISHMENTS

1.4.1 ATK Thiokol Formulation Development and Extrusion

Formulation Selection

A number of TP systems were identified that may allow twin-screw extrusion of MTTP in the specified temperature range of 80-120 °C. The ingredients in these TP systems are mostly common industrial polymers and plasticizers that are reasonably inert and nontoxic. Small mixes (10 g) of potential MTTP formulations containing these candidate TP systems were made in preparation for hazard testing. Since the hazard characteristics of these formulations were not characterized previously, the ingredients were mixed at ambient temperature as opposed to heating them to the actual processing temperature of 80-120 °C. Due to the ambient temperature mix, the ingredients of each TP system were predissolved in an appropriate solvent and were mixed as a slurry with the polytetrafluoroethylene (PTFE) and magnesium. Once the slurry was thoroughly mixed, the residual of the solvent was allowed to evaporate with intermittent stirring producing a material with a granular consistency. Teflon® 7C was selected as the grade of PTFE used in these mixes. Spherical magnesium meeting the military specification, Mil-M-382, Type III, Granulation 16 was also selected.

Samples of each "safety mix" were evaluated qualitatively for emission and burn time characteristics. A one-gram sample of each mix was ignited via a hot wire in an exhaust hood designated for burning small quantities of energetics. The following TP systems were eliminated for poor performance:

1. Microsere 5866, a microcrystalline wax derived from refining petroleum
2. Elvax 250, poly(ethylene-co-vinyl acetate) (hot glue ingredient)

3. Carnauba Wax (wax from palm tree exudate, a common ingredient in high quality car polish)

Hazards of the remaining formulations were analyzed. The data are reported in Table 1. The hazards were deemed acceptable for preparation of pellets to be used in radiometric measurements. It is noted that the autoignition temperature of PVC/GAP plasticizer formulation at 138 °C is somewhat low for extrusion at elevated temperatures. Also, it appears that liquid plasticizers mitigate MTTP friction and electro-static discharge (ESD) sensitivity. The TP systems containing THV220A do not contain a liquid plasticizer and exhibit a greater sensitivity to stimulus by friction. The only formulation with ESD sensitivity does not contain a liquid plasticizer. This suggests that TP systems with liquid plasticizers should be strongly considered as candidates for the ultimate MTTP composition especially considering the degree of sheer, a friction related stimulus, placed on a mix in a TSE.

Table 1. Summary of Hazards for Representative MTTP Formulations.

TP System	TP System Type	ABL Friction (lbs at 8 ft/sec)	Thiokol ESD (Joules)	Simulated Bulk Auto Ignition Test (SBAT), Onset (°F)	Thiokol Impact (inches)
PolyStyrene/DiMethyl Phthalate (PS/DMP)	ThermoPlastic (TP)/Plasticizer	800	>8	379	>46
THV220A (all PTFE replaced)	Thermoplastic FluoroPolymer (TFP)	130	>8	>500	>46
THV220A (PTFE present)	TFP (2THV220A:1 PTFE)	240	6.3 ± 2.4	>500	44.2 ± 0.9
THV220A/LFC-1	TFP/Fluoroplasticizer	180	>8	>500	>46
PVC/BenzylButylPhthalate (PVC/BBP)	TP/Plasticizer	800	>8	416	>46
PVC/GAP plasticizer	TP/Energetic Plasticizer	800	>8	282	>46

Twenty-gram hand mixes were made of the formulations whose hazard data are listed in Table 1 by the same method as was described for the 10-gram safety mixes. After the material was dried and granulated, pellets were pressed at ambient temperature in preparation for radiometric measurements. The pellets are 0.5” in diameter, have a mass of four grams and a height of approximately 0.75”. The pellets were coated with epoxy on the sides to inhibit burning on the sides of the pellet. This allows measurement of the burn time of the pellet since the pellet will burn in a uniform axial direction.

Placing B/KNO₃ granules on top of the pellet and igniting this first fire (FF) by a hot tungsten wire, in turn ignited each pellet. As the pellets burned, intensity versus time data were collected in regions of the near and mid IR (Figure 1) using a radiometer. The burn time in seconds/inch is measured as the difference in the time from when IR intensity rises to 50% of maximum intensity and when it falls back to 50% of maximum intensity divided by the pellet height. Integrating the area under the burn trace in the near IR yields the radiant energy (Joules/steradian) of interest for blackbody decoys. This can be normalized for comparison purposes by dividing the radiant energy by the weight of the pellet (Joules/steradian-gram). The radiant energy (not normalized) divided by burn time yields radiant intensity (Watts/steradian).

The initial radiometric and pellet burn time data collected during the program are summarized in Table 2. Pellets containing THV220A exhibited poor pellet integrity. In fact, formulations containing THV220A as the sole ingredient besides magnesium and those containing a mixture of THV220A and LFC-1 were severely cracked. These pellets ignited explosively. The formulation with THV220A and PTFE produced pellets with only small microfractures. Radiometric data were obtained on a few of these pellets. However measurements on others were hampered by rapid deflagration once the combustion front hit a fracture of significant size. Perhaps pellets with this TP could be produced without fractures at elevated temperatures. Further investigations of formulations containing THV220A were discontinued, however.

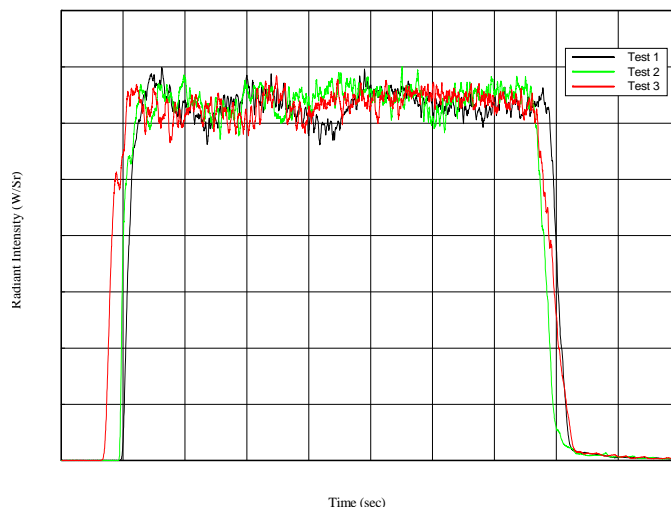


Figure 1. Burn Traces from MTTP Samples in the Near-IR Collected using a Radiometer.

Table 2. Summary of Initial Radiometric and Ballistic Data for MTTP Candidates.

Formulation	PTFE surface area	Mg Particle Type	% Mg	Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)
MTV (TSE)	Low	Spherical	54	3.1	315	185
PS/BBP	Low	Spherical	64	16.8	85	265
PS/DMP	High	Spherical	64	6.9	261	331
THV220A w/ PTFE	High	Spherical	60	8.1	171	261
PVC/BBP	Low	Spherical	65	17.1	67	215
PVC/BBP	High	Spherical	65	16.3	101	307
PVC/BBP	High	Spherical	60	15.3	118	330
PVC/GAP	High	Spherical	59	14.5	105	284
PVC/GAP	High	Spherical	54	14.7	115	316
PVC-co-PVAc/BBP	Low	Spherical	65	19.0	68	243
PVC-co-PVAc/BBP	High	Spherical	65	18.4	89	310

The MTV (TSE) formulation in Table 2 is a sample of the blackbody decoy material that MTTP is proposed to replace. Its burn time is 3.1 sec/inch. Acceptable burn times for MTV are reportedly in the range of 3-7 sec/in. The burn time of the samples containing THV220A with Teflon[®] 7C of 8.1 sec/in is quite close to the target burn time range. This suggests that if a suitable plasticizer for this TP can be found, this type of MTTP should have suitable radiometric and rheological properties. The PS/dimethylphthalate (DMP) formulation containing high surface area PTFE (Teflon[®] 7C) has a burn time that is barely within the acceptable range. The PVC formulations have significantly longer burn times and the burn times of formulations containing a copolymer of PVC with poly(vinyl acetate) are even longer. A significant aspect of the data for the PS, PVC and PVC-co-PVAc TP systems is that the normalized radiant energy is

higher than that for MTV. Thus, if means for shortening the burn time for this type of MTTP can be found, radiant intensity should be even higher than that for MTV at a given burn time. The slight improvement in burn time achieved by using the temperature-sensitive, very expensive energetic plasticizer, GAP, is not worth the risk of processing an MTTP containing it on a large scale. Burn times for formulations containing benzylbutylphthalate (BBP) were considerably longer than those for dimethylphthalate (DMP). These data were very instrumental in guiding the formulation refinement efforts that followed. Formulations containing the TPs, PS, PVC, and PVC-co-PVAc were selected for further refinement.

The materials dioctyladipate, (DOA, bis(2-ethylhexyl)adipate), and DMP, were evaluated seriously as plasticizers for the TP polymer. They lower the processing temperature at which MTTP can be processed. DOA is rather benign. DMP is benign with the exception that it is a potential fetotoxin and may affect fetal development. It is also subject to SARA Section 313 reporting requirements. Small samples of MTTP were mixed using solvent as described above in order to compare performance of formulations containing these two plasticizers. The type of plasticizer had little effect on the burn time of the MTTP formulation (Table 3). Unfortunately, DOA is not completely miscible in the TPs at the 1:1 TP/plasticizer ratio utilized in the selected MTTP formulations. When pellets of MTTP containing this plasticizer are pressed, residual DOA not absorbed by the respective TP is pressed out of the pellets. Thus, DMP was downselected as the plasticizer of choice. Other phthalates could be considered as replacements for DMP. However, they will most likely be identified as having comparable health risks once they have been scrutinized as closely as DMP. Care will be necessary while handling and disposing of this plasticizer. Table 4 provides a summary of the various plasticizers evaluated and an explanation as to why various candidates were eliminated.

Table 3. Comparison of the Effect on the Plasticizers, DMP and DOA on the Performance of MTTP Formulations.

Formulation	PTFE surface area	Mg Particle Type	% Mg	Burn Time (sec/in)	Radiant Intensity (W/sr)	Normalized Radiant Energy (J/sr-g)
MTV (TSE)	Low	Spherical (O)	54	3.1	315	185
PVC/DOA	High	50:50 O/chipped	60	13.0	129	308
PVC/DMP	High	50:50 O/chipped	60	14.1	111	293
PS/DOA	High	50:50 O/chipped	64	8.2	216	332
PS/DMP	High	50:50 O/chipped	64	9.0	180	301

Table 4. Summary of Plasticizer Selection.

Plasticizer	Why Eliminated?
Benzylbutylphthalate (BBP)	Longer burn times than some of the other plasticizers
GAP plasticizer	Expensive, temperature sensitive energetic plasticizer
Dioctyladipate (DOA)	Not completely miscible in the TPs at the 1:1 TP/plasticizer ratio utilized
Dimethylphthalate (DMP)	Selected as the plasticizer of choice

Attempts were made to shorten burn times of MTTP formulations by replacing some or all of the fine spherical magnesium with a higher surface area chipped magnesium (Figure 2). Generally speaking, the addition of chipped magnesium into the MTTP formulation did shorten burn times (Table 5) although some scatter in the data, presumably due to inconsistency in hand mixing the small samples, was evident. Higher surface PTFE also has a tendency to shorten MTTP burn times (Table 6). The higher surface area PTFE used was Whitcon TL-102.

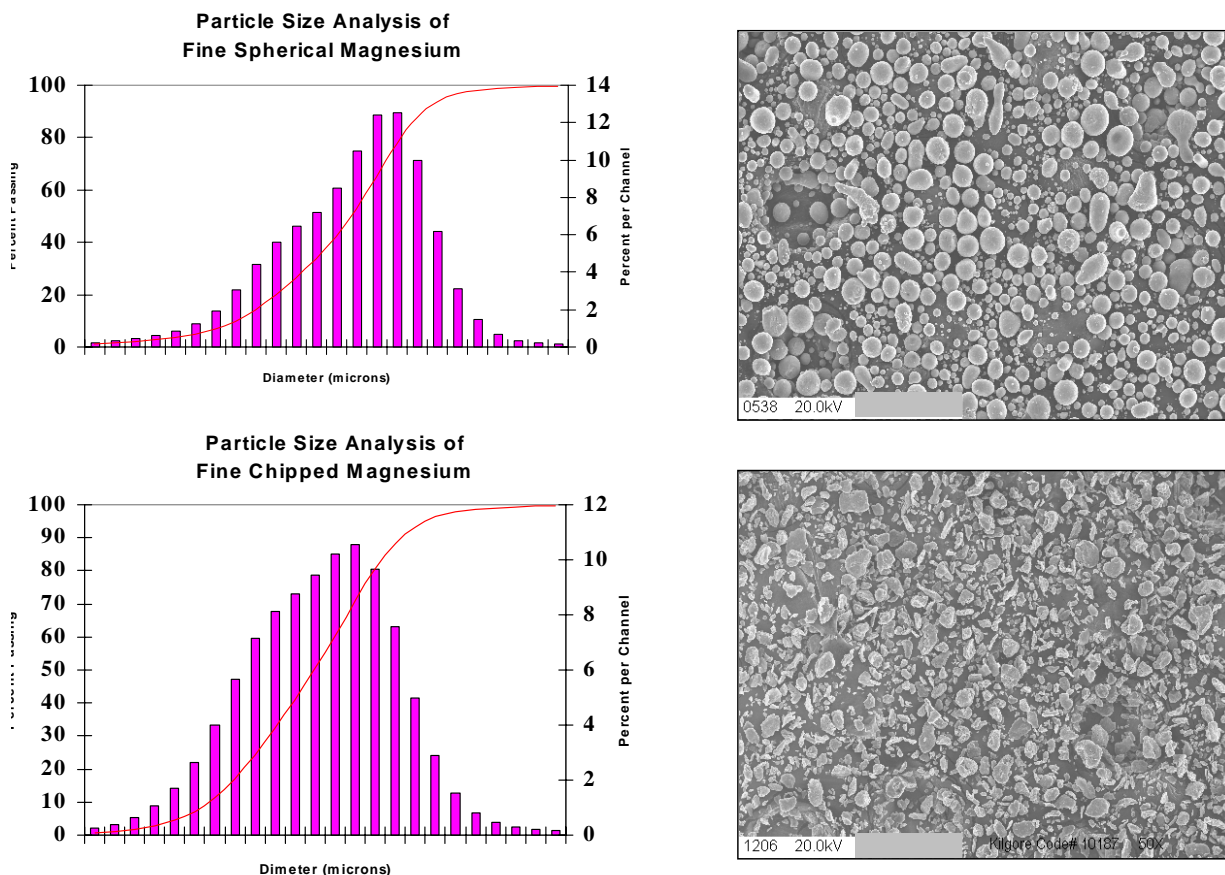


Figure 2. Comparison of Fine Chipped and Fine Spherical Magnesium used in the MTTP Formulations. The particle size distribution of the chipped magnesium (bottom left) is weighted towards smaller particle size relative to the fine spherical (top left). Its photomicrograph (bottom right) shows significantly higher surface area than the fine spherical magnesium (top right).

Table 5. Comparison of the Effect of Magnesium Surface Area on the Performance of MTTP Formulations.

Formulation	PTFE surface area	Mg Particle Type	% Mg	Burn Time (sec/in)	Radiant Intensity (W/sr)	Normalized Radiant Energy (J/sr-g)
MTV (TSE)	Low	Spherical (o)	54	3.1	315	185
PS/DMP	High	Chipped (-)	64	6.0	257	290
PS/DMP	High	50:50 O/-	64	9.0	180	301
PS/DMP	High	O	64	8.0	207	310
PS/DMP	High	O	64	6.9	220	284
PVC/DOA	High	50:50 O/-	60	13.0	129	308
PVC/DOA	High	O	60	16.9	97	308

Table 6. Comparison of the Effect on PTFE Surface Area on the Performance of MTTP Formulations.

Formulation	PTFE surface area	Mg Particle Type	% Mg	Burn Time (sec/in)	Radiant Intensity (W/sr)	Normalized Radiant Energy (J/sr-g)
MTV (TSE)	Low	Spherical	54	3.1	315	185
PVC/DOA	High	Spherical/chipped	60	13.0	129	308
PVC/DOA	Ultra-high	Spherical/chipped	60	12.2	135	308
PS/DOA	High	Spherical/chipped	64	8.2	216	332
PS/DOA	Ultra-high	Spherical/chipped	64	6.3	278	327

An experimental matrix was conducted investigating the effect of the four following combinations of ingredients on ballistic performance of MTTP formulations containing the PS/DMP plasticizer system (Table 7):

1. High surface area PTFE (Teflon[®] 7C)/fine spherical magnesium
2. Ultra-high surface area PTFE (Whitcon TL-102)/fine spherical magnesium
3. High surface area PTFE/fine chipped magnesium
4. Ultra-high surface area PTFE/fine chipped magnesium.

It is obvious that formulations with chipped magnesium have shorter burn times than those with spherical magnesium. Higher surface area PTFE promotes little or no difference in burn time of MTTP when it is mixed with spherical magnesium, but there is a marked decrease in burn time for formulations containing higher surface area PTFE mixed in the presence of chipped magnesium. Perhaps the sharp edges of the magnesium particles promote disaggregation of PTFE particles that, in turn, shortens burn times. It is important to reiterate that these formulations were mixed by hand on a small scale in the presence of solvent. The high shear, solventless environment of the TSE may promote disaggregation of the PTFE without the assistance of chipped magnesium. Selection of a PTFE was based on several criteria. Table 8 provides a short summary of both PTFE and the criteria behind selection or rejection.

Table 7. Results of an Experimental Matrix Varying Surface Area of both PTFE and Magnesium in MTTP Formulations.

Formulation	PTFE surface area	Mg Particle Type	% Mg	Burn Time (sec/in)	Radiant Intensity (W/sr)	Normalized Radiant Energy (J/sr-g)
MTV (TSE)	Low	Spherical	54	3.1	315	185
PS/DMP	High	Spherical	64	8.0	207	310
PS/DMP	Ultra-high	Spherical	64	8.4	201	317
PS/DMP	High	Chipped	64	6.0	257	290
PS/DMP	Ultra-high	Chipped	64	4.7	319	278

Table 8. Summary of PTFE Selection.

PTFE	Why eliminated?
Teflon® 7C (high surface area)	Difficulty feeding in available feeders, reduced performance versus other available PTFE
Whitcon TL-102 (ultra high surface area)	Selected as the Teflon® of choice

Processing concerns restricted the selection of acceptable magnesium for the formulation. As will be discussed in more detail later in this report, the extrusion process was limited to two feeders. As a result the decision was made to create a pre-blend of the binder, magnesium and catalyst as necessary. The viscosity of this feedstock greatly influenced the selection of an acceptable formulation. The major constraint associated with pre-blend viscosity was feedability. Many formulations resulted in material that was not capable of feeding from the 20-mm loss in weight (LIW). Table 9 provides performance summaries of two similar materials with the only difference between these formulations being in the make-up of the fine spherical magnesium, while all other variables are held constant. These magnesiums are designated ultra fine and fine. The formulation made with the ultra fine spherical magnesium (1863-70) was shown to out perform the fine spherical material (1943-25). Unfortunately the 1863-70 was not processable in the available feeders.

To determine the processability of proposed formulations, capillary rheometry and 20-mm LIW evaluations were performed on pre-blend formulations. Capillary work was also performed on the proposed full formulations. The necessity of creating a pre-blend presented several challenges that centered on the feedstock. While the formulation created with the ultra fine magnesium resulted in superior performance the necessary pre-blend was not processable. This material did not feed freely from the 20-mm LIW feeder. For the scope of the current program the use of ultra fine magnesium was abandoned. Figure 3 contains the capillary rheometry data that was used in determining the viscosity of the materials. The more viscous pre-blend was better suited for processing in the 20-mm LIW feeder. The fine spherical magnesium meeting military specification Mil-M-382C, Type III, Granulation 16 was down selected as the fine spherical magnesium of choice.

Table 9. Results of Candidate MTTP Formulations only Varying Fine Spherical Magnesium.

40.425% Spherical 100/200 mesh Mg, 17.325% fine spherical Mg, 9% OxyChem PVC-co-PVAc, 11.25% DMP, 18% Whitcon TL-102, 3% Sicotrans L2715D Fe₂O₃, 1% Aldrich 1-2 micron graphite				
	Fine Spherical Mg ID	Burn Time (s/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)
Vertical Batch Mix	(ultra fine) Mil-P-14067 Type I 200/325	7.9±0.3	189± 9	274 ± 4
Vertical Batch Mix	(fine) Mil-M-382C Type III Granulation 16 Special	13.1±0.1	110±5	256±8
19mm TSE	(fine) Mil-M-382C Type III Granulation 16 Special	11.4±0.9	120±24	241±32
MTV		3.1	315	185

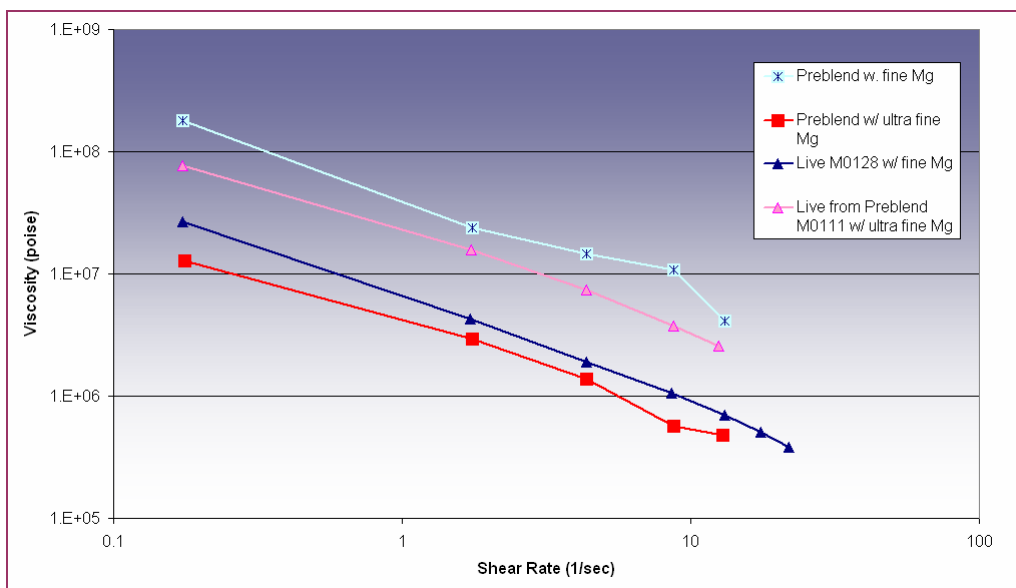


Figure 3. Capillary Rheology of Two Pre-Blends and their Resulting Full Formulations Containing Slight Different Magnesium Particle Size Distributions.

It was noted that two U.S. patents, # 4,981,534 and # 5,566,543, cite the use of plasticized PVC as a TP binder system in extrudable pyrotechnics to be used as the heat source in hybrid gas generants. The patents teach the use of ferric oxide as a burn rate catalyst therein. Iron oxide was added to the PVC and PS-based formulations (Table 10). As the percentage of iron oxide increased, the MTTP burn times decreased for formulations containing PVC. Also, as the surface area of the iron oxide increased, a decrease in burn time was observed. No significant effect on burn time was observed when iron oxide was added to PS-based MTTP formulations.

Table 10. The Effect of Iron Oxide as a Burn Rate Catalyst for MTTP Formulations Containing PVC.

Example	Percent TP	Percent Plasticizer	Percent PTFE	Percent Mg	Percent Burn Rate Enhancer	Normalized Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)
MTV	16% Viton A	0%	30% Algoflon	54% spherical (O)	0	3.1	315	185
Best PS/DMP	8% PS	8% DMP	20% Whitcon	64% chipped (--)	0	4.7	319	278
1	9% PVC	9% DOA	22% 7C Teflon	30%/30% O/--	0	14.1	111	293
2	9% PVC	9% DOA	21% 7C Teflon	30%/30% O/--	1% H-SA Fe2O3	7.1	217	289
3	9% PVC	9% DOA	19% 7C Teflon	30%/30% O/--	3% H-SA Fe2O3	4.0	307	232
4	9% PVC	9% DOA	19% 7C Teflon	30%/30% O/--	3% H-SA Fe2O3	7.3	214	294

While working with the TP systems, it became evident that PVAc-co-PVC (copolymer) had more desirable properties relative to thermal stability at elevated temperature and lower softening temperatures that made it more desirable as a TP in MTTP. Table 11 summarizes data showing that iron oxide also works efficiently as a burn rate catalyst in MTTP formulations containing the copolymer in the TP system. Again, as the percentage of iron oxide in the formulation increases, burn times decrease. Copper(II) based oxidizers in the form of finely divided powders were also investigated as MTTP burn rate catalysts. They exhibited catalytic behavior, but were not as effective as iron oxide catalysts.

Two formulations were downselected for evaluation with regard to processability in the TSE:

Polystyrene Based

64% Chipped Magnesium (Reade-RMC325)
 20% PTFE (Whitcon-TL102)
 8% Polystyrene (Huntsman T-817)
 8% Dimethylphthalate Plasticizer

PVC/PVAc Copolymer Based

60% Chipped Magnesium (Reade-RMC325)
 19% PTFE (Whitcon-TL102)
 9% PVC/PVAc Copolymer (Oxychem 1713)
 9% Dimethylphthalate Plasticizer
 3% Iron Oxide (BASF Sicotrans Red L2715D)

Table 11. The Effect of Iron Oxide as a Burn Rate Catalyst for MTTP Formulations Containing PVC-co-PVAc.

Example	Percent TP	Percent Plasticizer	Percent PTFE	Percent Mg	Percent Burn Rate Enhancer	Normalized Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)
MTV	16% Viton A	0%	30% Algoflon	54% spherical (O)	0	3.1	315	185
Best PS/DMP	8% PS	8% DMP	20% Whitcon	64% chipped (--)	0	4.7	319	278
5	9% copolymer	9% DMP	22% Whitcon TL-102	60% chipped	0	8.5	173	262
6	9% copolymer	9% DMP	19% Whitcon TL-102	60% chipped	3% H-SA Fe2O3	4.1	298	219
7	9% copolymer	9% DMP	18% Whitcon TL-102	60% chipped	3% H-SA Fe2O3 1% graphite	3.6	284	208
8	9% copolymer	9% DMP	16% Whitcon TL-102	60% chipped	6% H-SA Fe2O3	3.34	325	192

Thermoplastic polymers remaining in serious consideration as the TP for MTTP, polystyrene and the PVC/PVAc copolymer, are common polymers in industry. The greatest concerns environmentally are for the latter: the polymer typically contains residual (less than 1%) monomers, vinyl acetate and vinyl chloride, the former monomer is an animal carcinogen and is subject to SARA Section 313 reporting requirements.

There are also concerns with regard to combustion products derived from these polymers, especially the PVC/PVAc copolymer. An action item regarding analyzing for these combustion products was completed on the final MTTP formulation down selected (see below).

Since PVC/PVAc copolymer appears to have greater health risks and ecological concerns, it was to be selected only if there is a significant processing advantage, i.e., a lower temperature for acceptable processing using the TSE. Process safety is a major concern and must be weighed heavily in this development effort, as blackbody decoy composition undergoes very rapid deflagration at extremely high temperatures. Several polymer formulations were evaluated and eliminated for various reasons. Table 12 provides a summary of the polymers evaluated as well as an explanation for their elimination or down-selection.

Feedback from process studies necessitated formulations changes. In order for powdered Whitcon TL-102 PTFE to feed effectively, it must be blended with graphite. This formulation change actually shortened burn times in the case of the PVC/PVAc copolymer (Table 10). Additional plasticizer was added and a 70:30 mixture of coarse:fine spherical magnesium was used to promote processability. As discussed below, a formulation with the PVC/PVAc copolymer was eventually downselected for scale-up:

Magnesium, spherical, -100/+200 mesh	40.425%
Magnesium, fine spherical, Type III	17.325%
PTFE, Whitcon TL-102	18.000%
Dimethylphthalate	11.250%
PVC-co-PVAc, OxyChem 1713	9.000%
Iron Oxide, Sicotrans Red L2715D	3.000%
Graphite	1.000%

The selected formulation has a burn time of 13.1 sec/in, radiant intensity of 110 W/sr and normalized radiant energy of 256 J/sr-g. Because the selection of the final formulation was largely driven by processing factors, additional detail about the formulation selection will be included in the following sections.

Table 12. Summary of Thermoplastic Selection.

Thermoplastic	Why eliminated?
Microsere 5866	Poor performance when 1-gm sample ignited
Elvax 250	Poor performance when 1-gm sample ignited
Carnuba Wax	Poor performance when 1-gm sample ignited
THV220A	Eliminated due to poor pellet integrity and unstable ignition due micro-fractures that may have been over-come with a proper plasticizer
PVC	Eliminated because PVC co-polymer found to have more favorable properties such as thermal stability at elevated temperatures and lower softening temperature
Polystyrene	Eliminated due to poor initial TSE processing that may have been overcome
PVC-co-PVAc	Selected as the polymer of choice

Twin Screw Extruder Process Evaluation

Initially, it was intended to utilize the 58mm twin screw extruder (TSE) as the workhorse for compounding flare compositions containing TP binders in a continuous fashion. However, Kilgore Flares Company released a memo documenting recommendations for handling flare compositions, as a result of an accident that occurred in April 2001 while working with MTV flare composition, that influenced the intent to use the 58mm TSE. The recommendations from the Kilgore memo are as follows:

- “We believe that it is essential to remove the operators from the processing and drying of flare composition via automation, and where this is not possible, to limit the exposure to no more than the equivalent of two (2) pounds of dry composition. Where operators will be exposed to this amount of powder it will be in an appropriate pyrosuit. Should an incident occur, we expect no injuries to our operators, but possible damage to our equipment.
- We believe that it is essential in extrusion, pressing, coating and assembly to limit the amount of operator exposure to a single grain or pellet and to no longer use transport buggies for the storage and drying on in-process materials.

- We believe that administrative controls are to be minimized and used as a last resort when all other engineering controls are not feasible. We expect that the maximum hazard we will expose our operators to with the appropriate level of personnel protective equipment will result in an injury equivalent to less than a second degree burn”.

These recommendations prompted a decision to mix and compound in a much smaller TSE than initially intended. Processing efforts were re-directed from the 58mm TSE to the 19mm TSE, with SERDP approval. The major advantage of using a smaller extruder is that the amount of material in process is significantly reduced. For instance, typical throughputs for the 58mm TSE range from 100 – 200 lbs/hr. Throughputs for the 19mm TSE range from 2 – 6 lbs/hr. By using the 19mm TSE, the process would comply with industry safety concerns, specifically operator exposure to large quantities of flare composition, and still demonstrate concepts of continuous processing without the use of solvents.

Ultimately, a product collection system could be devised for the larger extruder, which would minimize operator handling, but design and construction of such a system is beyond the scope of this program.

Safety Protocol for Twin Screw Extrusion

Whenever new formulations are considered as candidates for continuous processing via twin-screw extrusion, specific protocols must be followed to ensure safety. Figure 4 shows the protocol followed for this program.

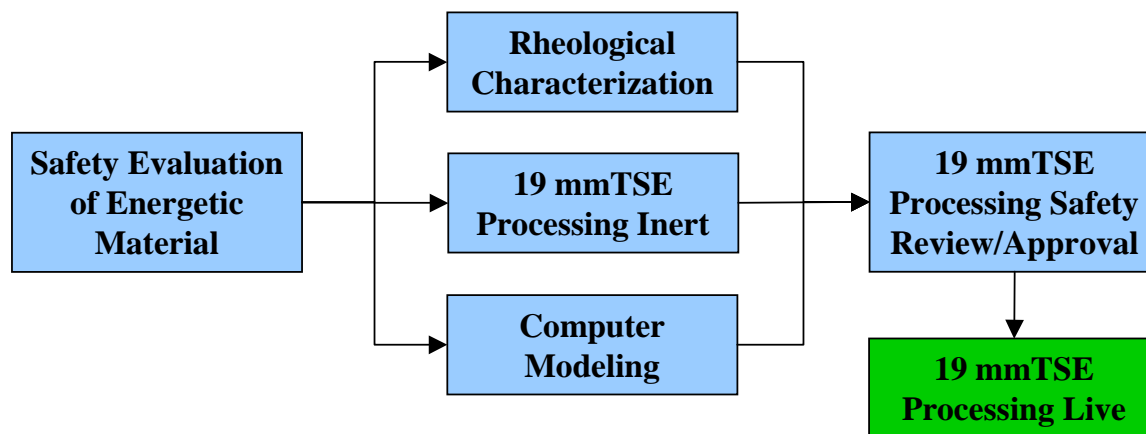


Figure 4. Extrusion Protocol.

A thorough safety evaluation of the formulation must be completed in order to understand the hazards and sensitivities of the materials involved. Rheological characterization, inert processing and computer modeling, provide valuable processing information. When all of the necessary requirements of the extrusion protocol have been accomplished, the process is reviewed to ensure safety before operating with energetic materials. In order to generate data to support the safety review several tasks are required. The major tasks are as follows:

- Standard safety testing for potential formulations and intermediates
- Feed system evaluations to characterize potential feed streams
- Rheological characterization of potential formulations and intermediates
- Inert evaluation

Standard Safety Testing

The standard safety tests are designed to identify sensitivities associated with impact, friction, ESD, temperature, and detonation susceptibility of the potential formulations to be processed. Two general formulations have been down selected as potential candidates based on ballistic performance as described above. Each utilize a different binder, one uses a polystyrene system the other uses a PVC-co-PVAc system. Standard safety tests have been performed on these two families and are shown below in Table 13.

Table 13. Standard Safety Test Data for General Formulations.

Formulation Family	Required Safety Tests				
	Thiokol Impact (in)	ABL Sliding Friction		ESD (Joules)	SBAT (°F)
		Load (lbs)	Rate (ft/sec)		
Polystyrene	> 46	800	8	>8	371
PVC-co-PVAc	> 46	800	8	>8	358

The safety data for each family are very similar. These data do not identify any unusual ignition sensitivities. Additional safety testing was performed including Russian DDT to determine detonation susceptibility on the actual formulation selected for processing.

Table 14. Safety Data of Full Formulation and Intermediates.

Material Name	SBAT onset Temp °F	TC ESD, Unconfined (J)	ABL Friction (lbs) @ 8ft/s	TC Impact (in.)	Russian DDT @ 500 psi
Mg (Milm-382c Gr 16 Special)	No Reaction/ No Burn	1.5 J Mass Ignition @ 8J	800	>46	“No Go” No Report
Mg (-100/+200)	No Reaction/ No Burn	>8	800	>46	Not Tested
Mixed Pre-Blend	327	6.9	800	>46	“No Go” No Report
Fitz-Milled Pre-Blends	342	7.5	800	>46	“No Go” No Report
Full Formulation	348	>8	800	>46	“No Go” Slight Report

Once the formulation had been selected additional safety testing was performed. Not only were safety tests performed on the full formulation material, but extensive safety testing was also performed on several of the intermediates. These intermediates included the pre-blend (in the mixed form and as the material was prepared for feedstock) and the different forms of the magnesium. The results of these tests are included in Table 14.

Feed Stream Development

In addition to reduced throughputs mentioned above, there were other tradeoffs associated with switching from the 58mm TSE to the 19mm TSE that impacted the original processing approach. For example, the 58mm TSE system is equipped with three solid LIW feeders and two liquid feeders. The 19mm has one solid LIW feeder, one solid volumetric feeder and one liquid feeder. Both potential formulations contain more ingredients than the 19mm extrusion facility could accommodate as individual feed streams, therefore, they needed to be combined in some fashion. Two possible approaches emerged as feasible candidates for processing MTTP, a multiple pass approach and a single pass approach.

Multiple Pass Approach

A schematic of the multiple pass approach is shown in Figure 5. The two formulation families are similar to each other with respect to the types of raw materials and formulation percentages, therefore, feed stream evaluations used polystyrene as the baseline. As shown, the multiple pass approach required multiple passes of some materials through the extruder. The initial pass combines the binder, plasticizer, and magnesium together and incorporates Teflon® during the final pass through the extruder.

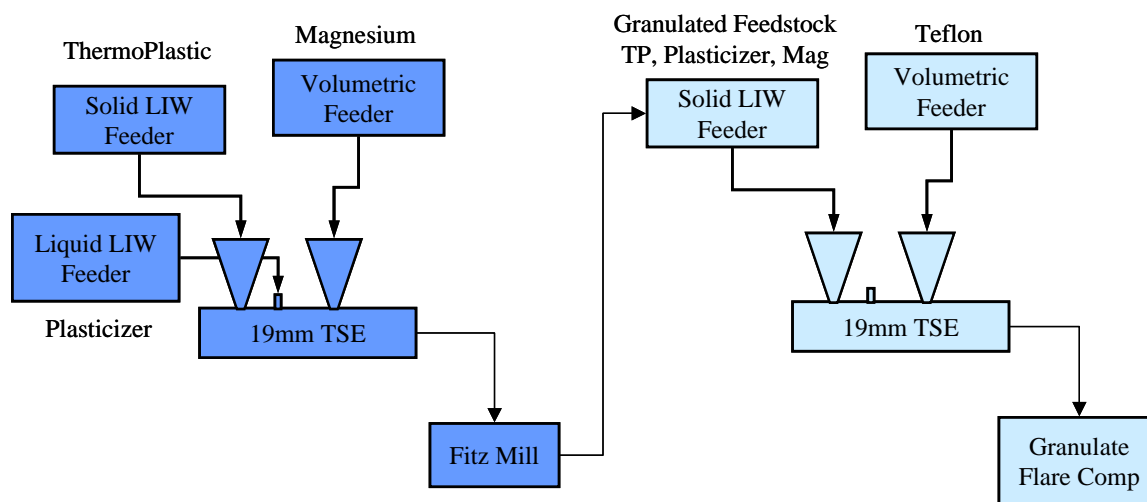


Figure 5. Multiple Pass Approach for Processing MTTP.

As mentioned, the maximum throughput for the 19mm TSE is in the neighborhood of 6 lbs/hr. Some constituents in the formulations are relatively small percentages, 8 to 10 percent, which caused some concerns with the capability of the feeders: the multiple pass approach required the LIW feeder to feed at extremely low rates. To determine feeder capability, systematic feed studies were performed. The raw material feed rates were established based on a throughput of 5.8 lbs/hr through the extruder and using a ballpark formulation of 10% binder, 10% plasticizer, 60% magnesium and 20% Teflon®. The polystyrene feed stream calculated to be 0.72 lbs/hr. This rate was examined first to determine if the LIW feeder could perform properly at such a low rate. Results of the feed test are shown in Figure 6.

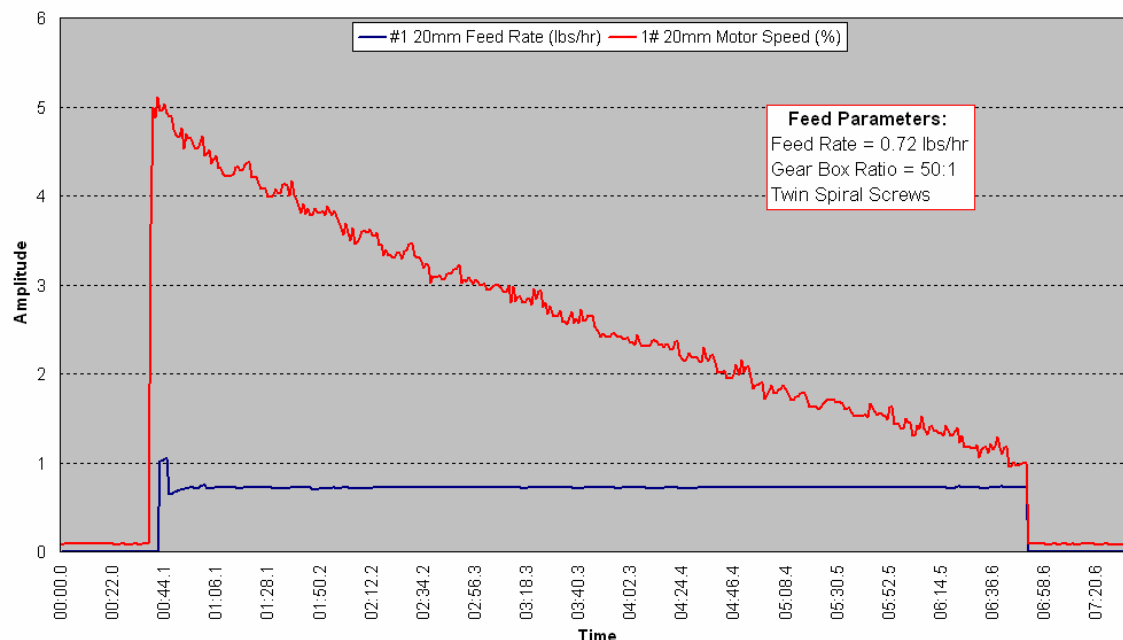


Figure 6. Initial Polystyrene Feed Test with Twin Spiral Screws.

During operation, the feed system tried to find the proper motor speed to deliver the appropriate amount of material, however, the feed system never stabilized and shut down within minutes due to low motor speed. The plot identifies the range at which the motor was trying to operate in order to meet the target set point of the feeder. Typically, feeders should be operated around 50% motor speed for optimal operation. This allows the system to adjust the motor speed up or down as required. For example, if the bulk density of the material should increase or decrease as a result of segregation while feeding, the feed system will increase or decrease the motor speed to maintain the set point delivery rate. If the system is operating near the minimum or maximum capability, formulation deviations could result.

One way to adjust or shift the operating range of the feed system is to change the type of feed screws. To determine the relationship of feed screws to motor speed, the original feed screws were swapped with a different set and the feed test repeated. The feed screws used for each feed test differed significantly. The screws for the first test were twin spiral screws that do not intermesh, much like an auger type screw. The second feed test used concave intermeshing screws that have much tighter clearances than the twin spiral set. Similar results were realized as shown in Figure 7.

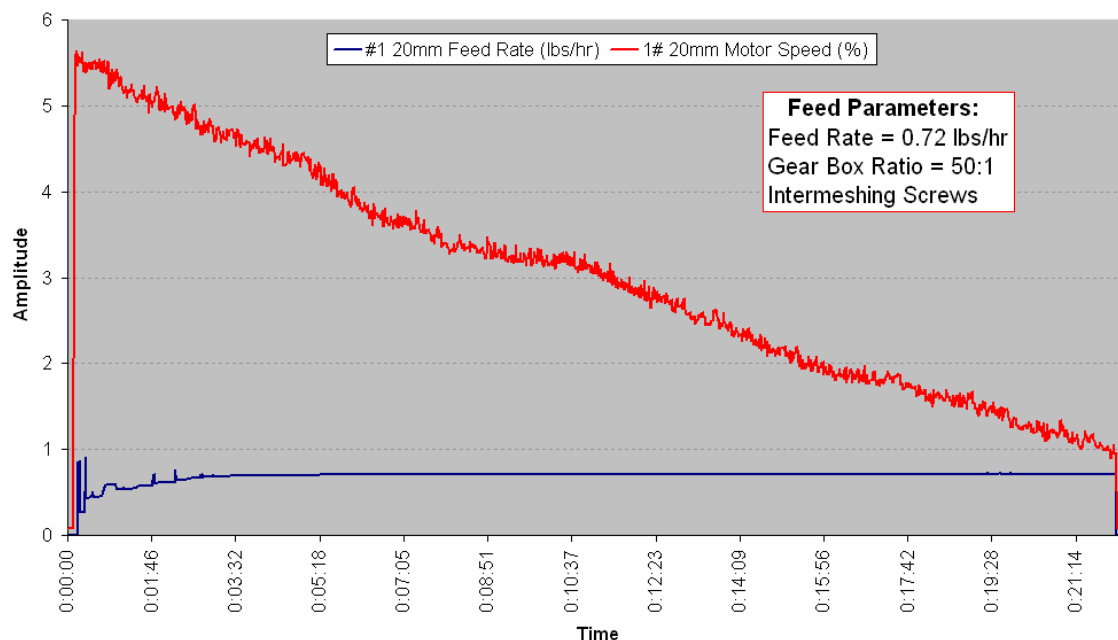


Figure 7. Polystyrene Feed Test Using Concave Intermeshing Screws.

The feeder ran a bit longer, but the range of operation did not change. Based on the initial feed stream evaluations, it became obvious that the LIW feeder could not control at such low feed rates. One solution to the problem is to increase the amount of binder in the formulation at the expense of another constituent. For instance, remove all or some portion of the plasticizer and replace it with binder, in this case, polystyrene. Using this approach, a single feed study was conducted where all of the plasticizer was sacrificed and replaced with polystyrene. Based on extruder throughputs of 5.8 lbs/hr, the feed rate of the polystyrene doubled from 0.72 lbs/hr to 1.44 lbs/hr. The results of the feed study with increased binder concentration revealed that the feed system stabilized and delivered the 1.44 lbs/hr to the extruder for compounding, however, the operating range did not shift dramatically. The data from the feed test are shown below in Figure 8.

On a positive note, the motor speed did operate in a very tight range over a time period of approximately 30 minutes, which indicates the feed system did not need to make significant adjustments to the motor speed as a result of perturbations to the system. Therefore, inert extrusion runs were conducted using only polystyrene to determine how it would behave in the extruder without plasticizer present, and to begin to establish the operating parameters of the extruder including screw configuration, screw speed, and process temperatures.

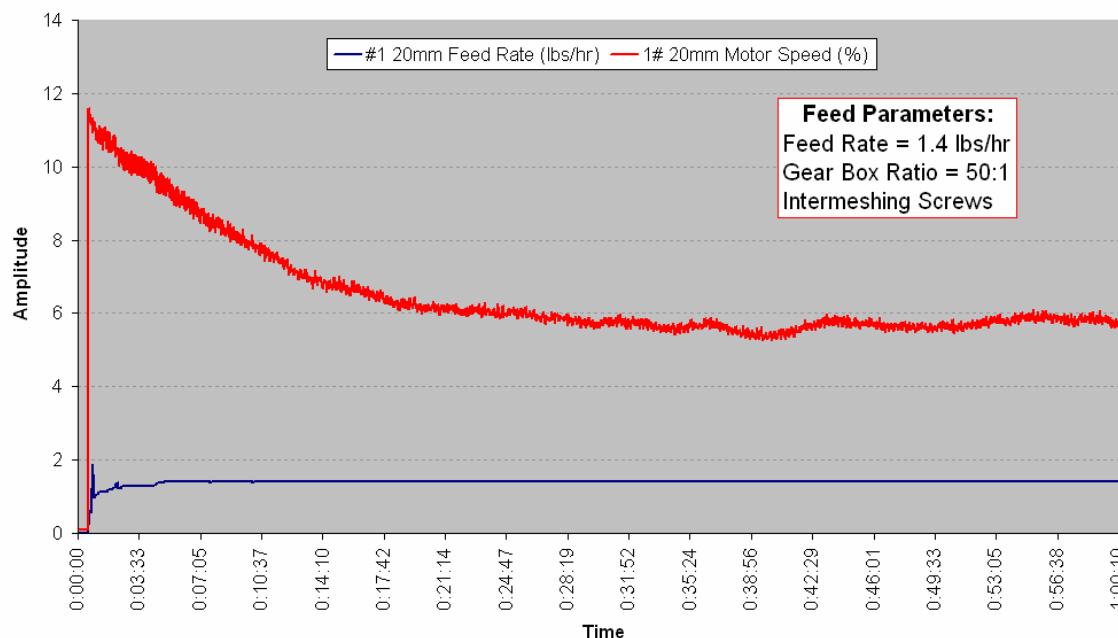


Figure 8. Polystyrene Feed Test at Increased Throughput.

The first attempt at processing polystyrene in the extruder showed promise. The screw configuration for the extruder was based on a previous work done in the 19mm TSE. The processing temperatures for each of the barrel sections were set at 240°F due to the melt point of polystyrene and for safety reasons. As a general safety rule when processing energetic materials, operating temperatures should be at least 100 degrees below the auto-ignition temperature. For these types of materials, the auto-ignition temperatures range from 340°F to 380°F. This methodology maintains a safe margin for operation while processing. The extruder screws ran at 65 rpm for the evaluation. The LIW feeder fed polystyrene into the extruder at a rate of 1.44 lbs/hr as established from previous feeding tests. Post extrusion examination revealed that the polystyrene did not melt in the region designed to melt it. In fact, the material did not melt until the very end of the extrusion cycle. Figure 9 shows the progression of the material as it traveled along the screws of the extruder.

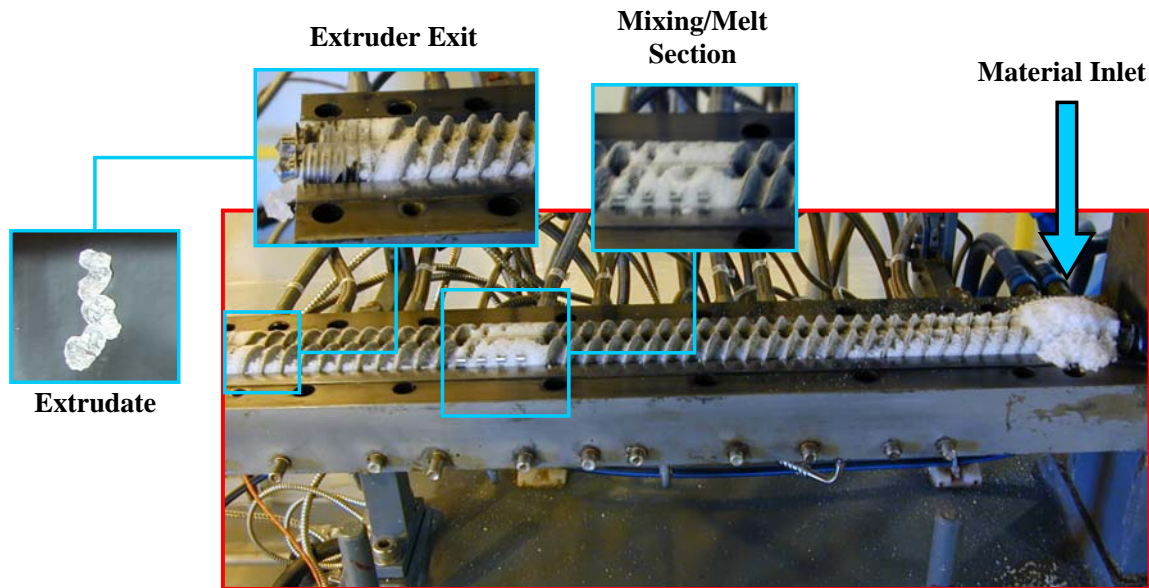


Figure 9. Initial Extrusion of Polystyrene.

At the material inlet, material falls onto conveying screw elements that move it along to the melting section. As material enters the melting section, it slows down, increasing the local residence time and allowing the material to heat and eventually melt. Specially designed screw elements called kneading blocks impart a significant amount of shear to the material in this region. At this point in the process, the material needs to be melted and fluid so that other materials can be added easily further downstream. However, the material did not melt as intended, indicating that the design of the screw was not aggressive enough to melt the material before leaving the melt zone. Once material leaves the melt zone, it is conveyed downstream to the end of the extruder. At the end, smaller pitched elements slow the material down once again. The material did eventually melt as shown in the figure, demonstrating that the material could be melted under the right circumstances. Additional inert runs of polystyrene evaluated other screw designs. These runs demonstrated that the material could be melted in the intended region but created additional concerns that were not apparent before hand. During extrusion, premature shutdown of the process occurred due to excessive torque developed in the extruder. Data from the run are shown in Figure 10. These levels of torque are not favorable and need to be avoided. Post examination of the extruder showed that the screw fill in the melt section increased significantly, indicating that the residence time had increased. However, the fill increase caused the torque to increase as well. Figure 11 shows pictures of the extruder immediately after shutdown.

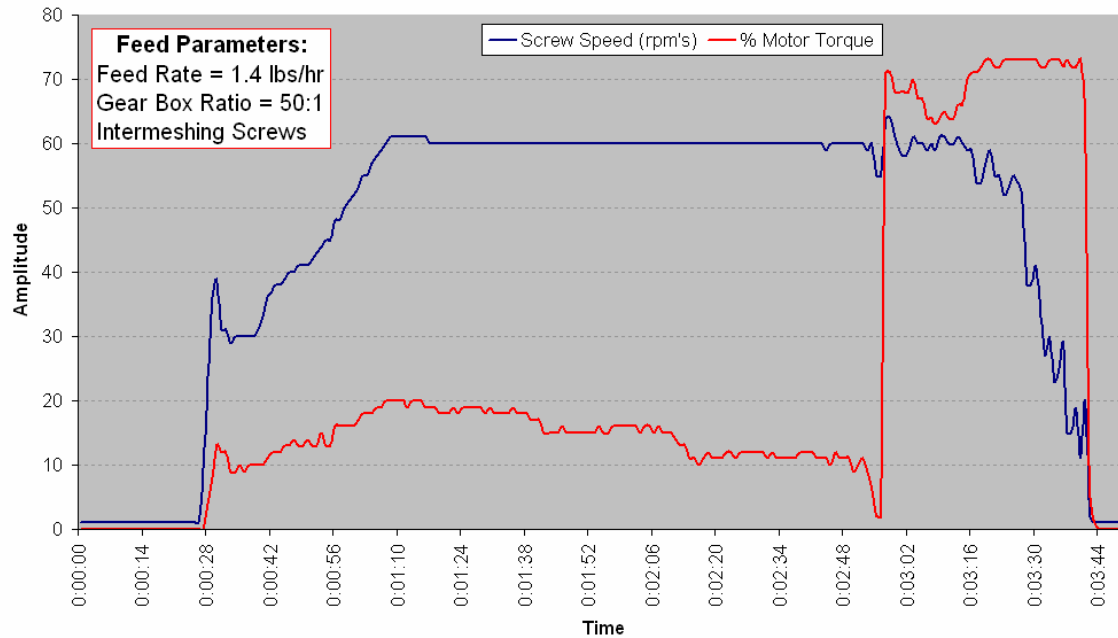


Figure 10. Extrusion Data for Polystyrene with Modified Screw Design.

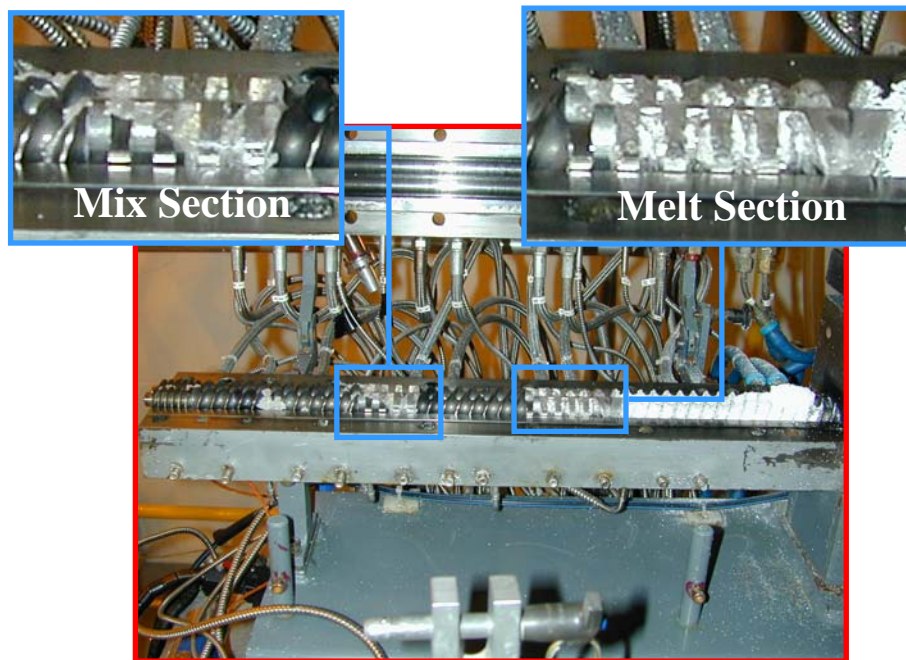


Figure 11. Post Extrusion of Polystyrene with Modified Screw Design.

A limited amount of material passed through the melt section and into the mix section, but the melt section restricted the majority of it from passing through.

The two inert evaluations reported above used screw designs at each end of the spectrum, a mild screw design and a very aggressive screw design, to determine how polystyrene would behave in the extruder without the addition of plasticizer. Polystyrene without plasticizer could not be extruded at the operating parameters of the extruder due to its high viscosity. Melt viscosities of TPs are inversely proportional to temperature: as temperature increases, viscosity decreases. Processing temperatures are restricted due to safety concerns and cannot be increased to lower the viscosity of the TP. Since the melt temperatures are similar for polystyrene and PVC-co-PVAc, based on these data, the binder system requires plasticizer to lower the melt temperature and decrease the viscosity for processing.

In summary, the multiple pass approach for processing MTTP formulations was evaluated by means of systematic feed studies and inert extrusion runs. The data did not validate the multiple pass approach because of the capability of the LIW solid feeder and the behavior of the binder without plasticizer in the extruder. Work efforts therefore shifted to evaluate the single pass approach.

Single Pass Approach with Polystyrene

Figure 12 shows the schematic for the single pass approach. The single pass approach uses a feedstock prepared in a vertical batch mixer. The mixer combines raw material ingredients to reduce the number of feed streams required and also eliminates the need to feed small quantities from the LIW solid feeder. The binder, plasticizer, and magnesium were logical choices for creating a feedstock, protecting the Teflon[®] from exposure to unnecessary work required to heat and melt the binder.

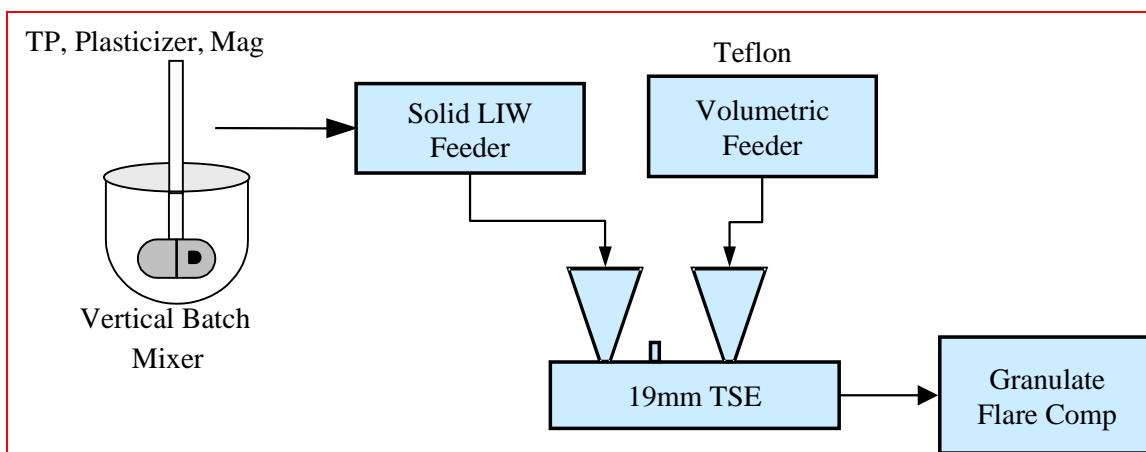


Figure 12. Single Pass Approach for Processing MTTP.

Several variations of pre-mixed materials were evaluated as potential feedstock using qualitative, capillary, feeder, and TSE assessments of the pre-mixed materials. Table 15 summarizes the results of the evaluation. Polystyrene blends appeared to be the most promising, therefore, many combinations containing different plasticizer to binder ratios, magnesium shapes and ratios were evaluated prior to examining PVC-co-PVAc systems.

Table 15. Summary of Various Pre-Blend Feedstock Materials.

Mix #	P/TP Ratio	Ingredients	%	Qualitative Assessment		Capillary Assessment		Feeder Assessment		TSE Assessment	
				Go	No-Go	Go	No-Go	Go	No-Go	Go	No-Go
1863-46	1.0	Polystyrene	10	Granular			X	4 lbs/hr @ 30% motor sp.			Over Torqued
		DMP	10								
		Chipped Mag	80								
1863-66	1.25	Polystyrene	10		Material too sticky to feed	NA	NA	NA	NA	NA	NA
		DMP	12.5								
		Fine Spherical (Sph.) Mag	23.25								
		Chipped Mag	54.25								
1863-67a	1.125	Polystyrene	10		Material too sticky to feed	NA	NA	NA	NA	NA	NA
		DMP	11.25								
		Fine Sph. Mag	39.375								
		Coarse Sph. Mag	39.375								
1863-67b	1.125	Polystyrene	10	Granular			X	4 lbs/hr @ 30% motor sp.			Over Torqued
		DMP	11.25								
		Fine Sph. Mag	39.375								
		Chipped Mag	39.375								
1863-68	1.125	Polystyrene	10	Granular			X	4 lbs/hr @ 30% motor sp.			Determined to be not processable (1863-67b)
		DMP	11.25								
		Fine Sph. Mag	19.6875								
		Chipped Mag	59.0625								
1863-70	1.25	PVC/PVAc	11.11	Granular		X		4 lbs/hr @ 40% motor sp.		70 rpm ~24% torque, material extremely soft	
		DMP	13.89								
		Ultra Fine Sph Mag Type I	21.39								
		Sph Mag 100/200 mesh	49.91								
		Iron Oxide	3.7								
1943-25		PVC/PVAc	11.11	Granular		X		1.8 lbs/hr @ 19% motor sp.		90 rpm ~31% torque, material extremely soft	
		DMP	13.89								
		Fine Sph Mag Type III	21.39								
		Sph Mag 100/200 mesh	49.91								
		Iron Oxide	3.7								

Each of the pre-blended feedstock materials were screened in some fashion, if possible, to ensure the material would feed. Many of the potential combinations were eliminated immediately from a qualitative perspective.

Ballistic performance gathered from subscale mixes demonstrated that the best polystyrene formulation contained 8% polystyrene, 8% DMP (plasticizer), 20% Teflon[®], and 64% chipped magnesium. This formulation has a 1:1 plasticizer to binder ratio. A pre-blended feedstock was made in a vertical mixer compounding polystyrene, DMP, and magnesium to produce a homogenous blend designated as mix 1863-46. The material did not stick together and was very granular, which allowed it to flow very easily. Feed tests conducted using this material showed that the material fed extremely well. The feed rate for the pre-blended material targeted 4.0 lbs/hr. A picture of the material and the results of the study are shown in Figure 13. The data show that the motor speed increased significantly and stabilized near 30% to maintain the set point feed rate.

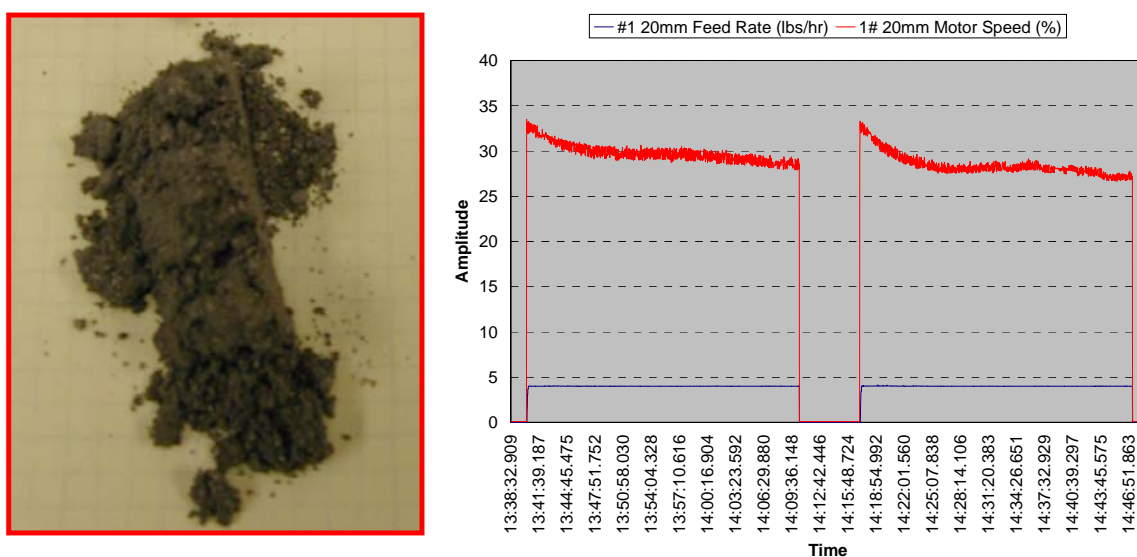


Figure 13. Pre-Blended Material and Feed Study Results for Mix 1863-46.

Following the feed studies, the material was analyzed with the capillary rheometer and 19mm TSE to get a feel for the rheology of the material and its behavior in a high shear environment. These experiments were conducted in parallel. The capillary rheometer is a ram extruder that uses a variety of capillaries to create a range of shear rates analogous to the shear environment in the TSE. The pre-blended material did not extrude through any of the capillaries, but simply compacted into a slug as shown in Figure 14. The TSE experiment produced very similar results in the melting section of the screw. Figure 15 shows the melt section of the screw and the extrusion data. When the pre-blended material filled the melt region, the torque increased dramatically and eventually shutdown the process. Both the capillary and TSE evaluations revealed that this pre-blend, 1863-46, did not have adequate rheology conducive to ram or twin screw extrusion.

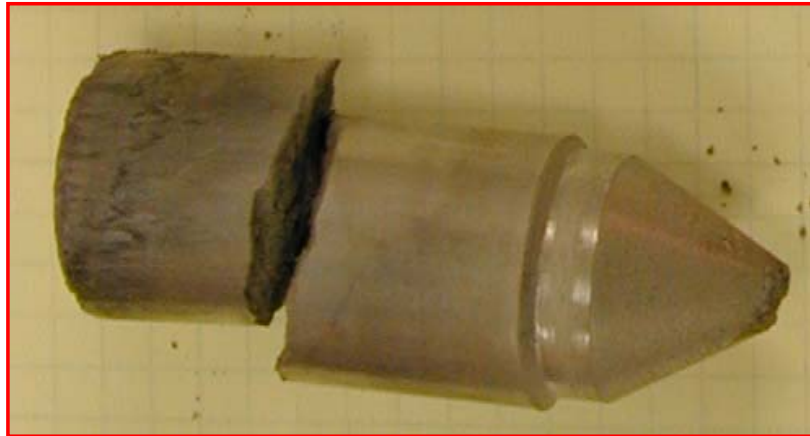


Figure 14. Capillary Results with 1863-46 Pre-Blended Material.

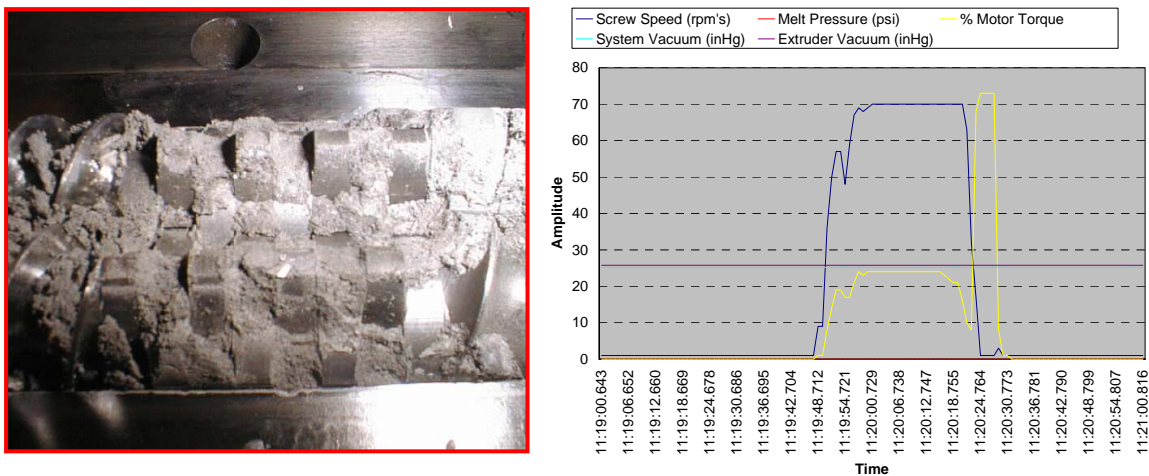


Figure 15. Melting Section of TSE and Extrusion Data for Mix 1863-46.

Pre-blend mixes 1863-66 and -67a varied the plasticizer to binder ratio and magnesium shape and ratio. Neither of the pre-blends were granular like mix 1863-46, in fact, both agglomerated making one large single piece of material. Mix 1863-67a had a lower plasticizer to binder ratio and a different combination of magnesium. It looked slightly drier than mix 1863-66, however, neither of the mixes could be processed. Figure 16 shows a picture of each pre-blend.



Figure 16. Pre-Blend Mixes 1863-66 and 1863-67a.

Single Pass Approach with PVC-co-PVAc

All of the other polystyrene pre-blend mixes listed in Table 15 exhibited characteristics that made them unprocessable in some form or another, therefore, work efforts shifted to MTTP formulations containing a PVC-co-PVAc binder system. Pre-blend mix 1863-70 contained PVC-co-PVAc as the binder, with a plasticizer to binder ratio of 1.25. The material was very granular following Stokes granulation, as shown in Figure 17, and flowed easily.



Figure 17. PVC-co-PVAc Pre-Blend Mix 1863-70 following Stokes Granulation.

The material fed very well in the LIW solid feeder at a target feed rate of 4.0 lbs/hr. The motor speed stabilized near 42 %, which is optimal for feeding. The feed test lasted approximately 35 minutes. The motor speed varied very slightly while in operation, indicating that no significant perturbations occurred within the feed system. The feed data are shown in Figure 18.

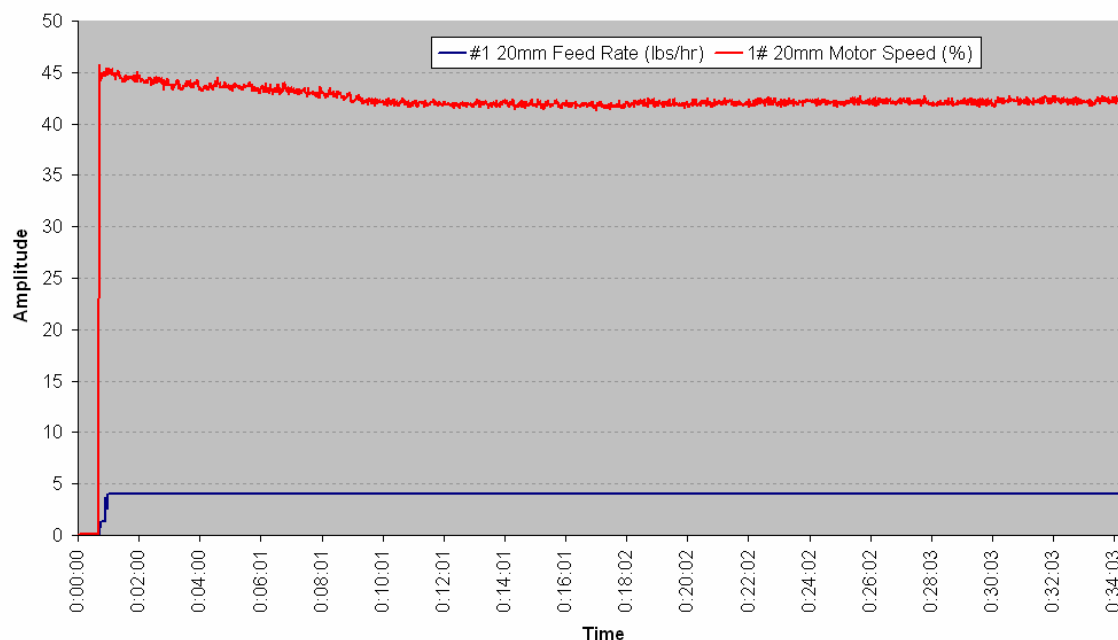


Figure 18. Feed Test Data for PVC-co-PVAc Pre-Blend Mix 1863-70.

This material extruded through a capillary easily, therefore, multiple runs were made at different temperatures to verify the relationship between viscosity and temperature, and to determine the optimum extrusion temperature for the TSE. Figure 19 compares viscosities at two distinct temperatures, 220 °F and 240 °F. The apparent viscosity of the material changed significantly from one temperature to the other. These data infer that at 220 °F, the material is not totally melted. A higher temperature was tried to see if increasing the temperature further would decrease the viscosity even more, however, higher temperatures did not lower the viscosity any further. The data are shown in Figure 20. Based on initial capillary data for the PVC-co-PVAc pre-blended feedstock, the optimal processing temperature for TSE is 240 °F.

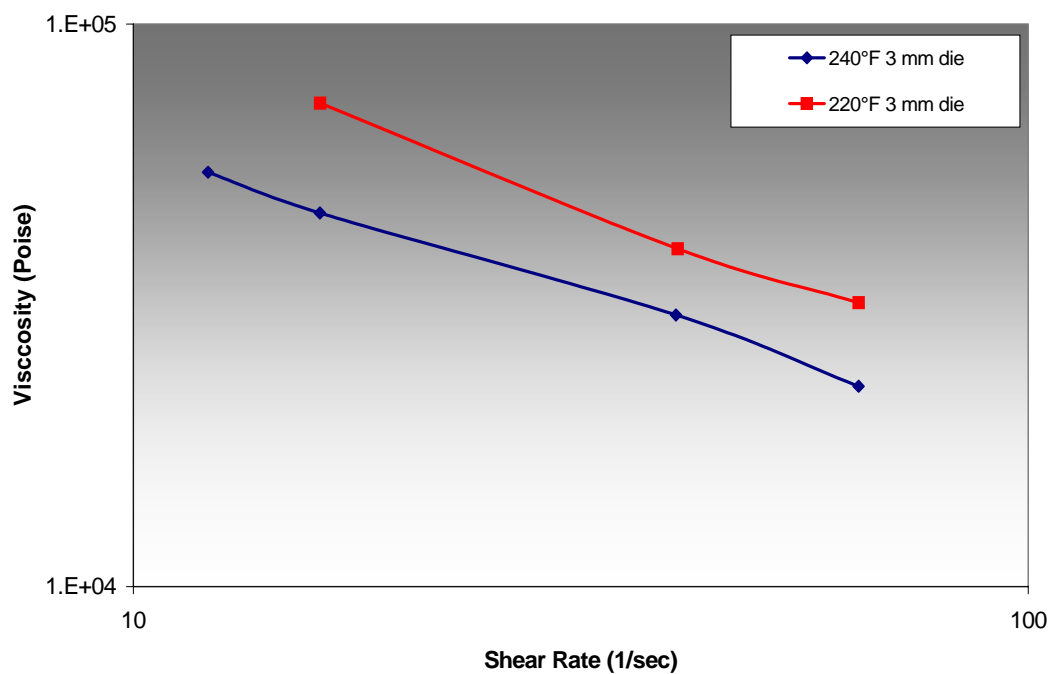


Figure 19. Viscosity Plots for Pre-Blend Material Mix 1863-70.

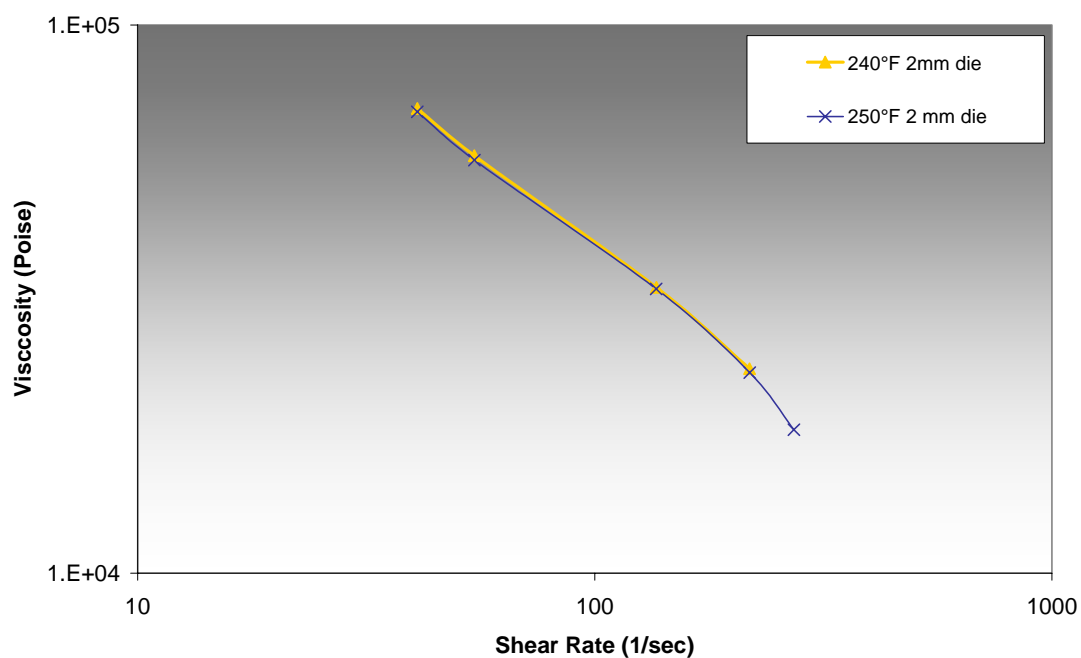


Figure 20. Additional Viscosity Data for Pre-Blend Material Mix 1863-70.

Following the selection of an optimum processing temperature further evaluation of this pre-blend and its resulting full formulation or ‘live’ material was completed. The 1863-70 based

‘live’ formulation (designated as 1863-71) provided the necessary radiometric properties needed. This formulation contained a coarse Mg (-100+200 mesh) and an ultra fine Mg (-325 mesh). In May 03, rheological properties were run on 1863-70 pre-blend material (Mix # M0111), ‘live’ material (Teflon[®] and graphite added to pre-blend mix# M0111), and an inert simulant (bicarbonate and graphite added to pre-blend mix# M0112). Mixes were prepared in a vertical mixer. The results of capillary extrusion of these materials are summarized in Figure 21.

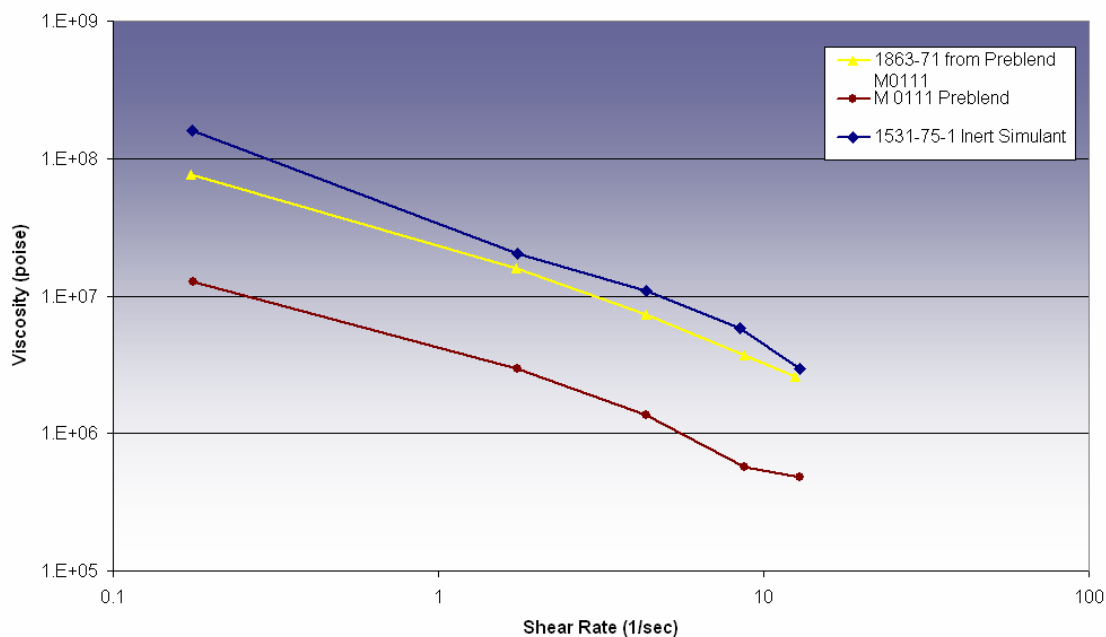


Figure 21. Apparent Viscosities of 1863-70 based Formulations.

No noticeable problems were seen with any of the materials. It was expected that the apparent viscosity of 1863-71 material was much greater than that of the 1863-70 pre-blend due to the higher degree of solids. The inert stimulant had a slightly higher viscosity than 1863-71 material, which is advantageous, as it meant if the inert could be successfully processed in the 19mm TSE without experiencing high torque levels, the ‘live’ material would process at lower torque levels.

Results from TSE efforts were also encouraging for this formulation. A short inert extrusion run processed in the 19mm TSE using pre-blend mix 1863-70 was successful. The operating parameters for the run were: screw speed of 70 rpm, barrel and die temperatures of 240 °F, and a throughput of 4lbs/hr. As the material filled the screws of the extruder, the machine torque stabilized at 24%, which is well within the acceptable torque range of the equipment.

A plot of the data for the extrusion run, pictures immediately following termination, and of the extrudate are shown in Figure 22, Figure 23, and Figure 24, respectively.

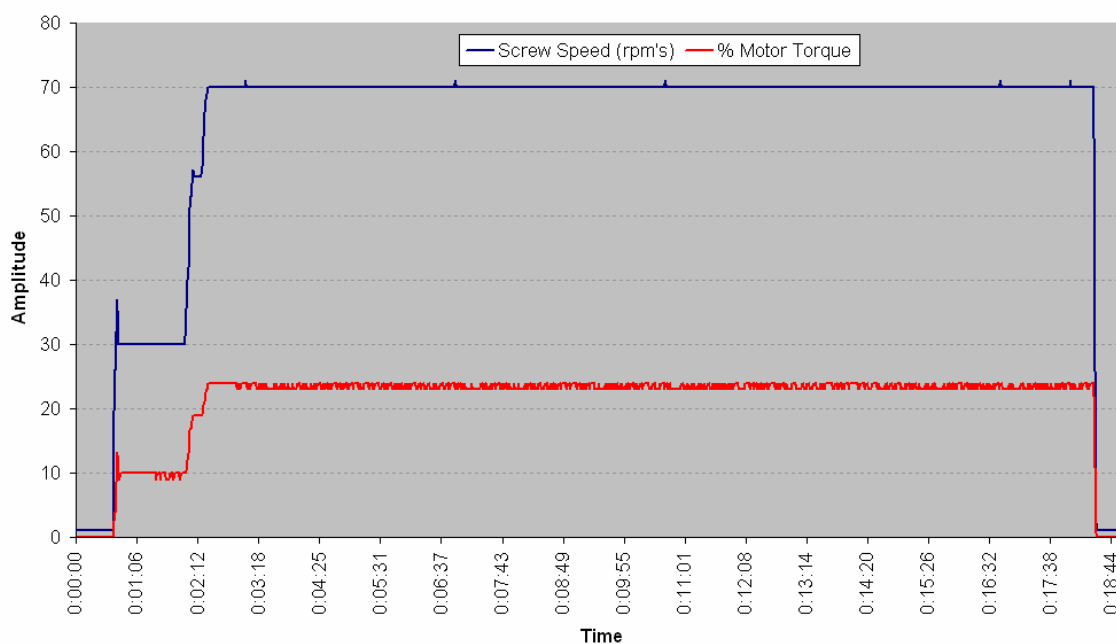


Figure 22. TSE Data for PVC-coPVAc Pre-Blend Mix 1863-70.

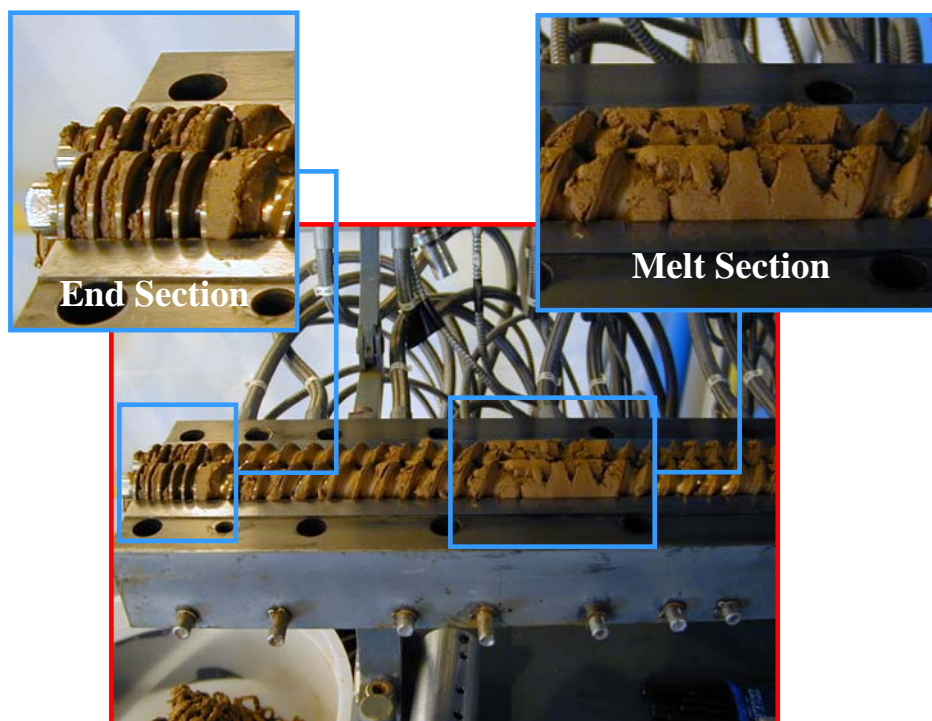


Figure 23. Post Extrusion of Mix 1863-70.



Figure 24. Pre-Blend 1863-70 Extrudate from TSE.

The material remained very soft and pliable once melted inside the melt section. Teflon[®] could easily be incorporated into the melted material. As it exited the extruder, the material broke off in the form of ribbons and did not stick together afterwards.

Following the positive demonstration of pre-blend 1863-70 further processing evaluations were required. With this increased demand for pre-blend came the desire for to be able to process the material on a larger scale. To accomplish this it was determined that a more effective method for granulating the pre-blend was necessary. As a result it was desirable to take the mixed pre-blend to the Fitz-mill versus granulation in the Stokes. 1863-70 material was taken to the Fitz-mill and produced a feedstock that looked very promising, though not consistent with the granulation seen in the Stokes. Unfortunately, when this material was taken to the 20-mm LIW feeder it was not able to feed properly. Due to processing constraints it was determined that substituting the ultra fine spherical magnesium for magnesium that was fine spherical may alleviate the problem. This new pre-blend formulation was designated 1943-25.

The new MTTP pre-blend, 1943-25, was taken to the 20-mm LIW feeder and was shown to feed with no difficulty. Further processing efforts revolved around the use of this pre-blend as the cornerstone of the formulation.

In Jan 04, a mix of the 'live' formulation with the larger particle size Mg (based on 1943-25) was made in the vertical mixer and compared to the original baseline material, 1863-71 (see Figure 25). As can be seen, the addition of a larger particle size material reduces the viscosity significantly. The apparent viscosity of the material made with fine Mg is approximately 3 times less than the formulation with the ultra fine Mg. Due to the more favorable capillary demonstration on the ability to effectively feed 1943-25 pre-blend in the 20-mm LIW feeder, it was selected as the baseline MTTP formulation.

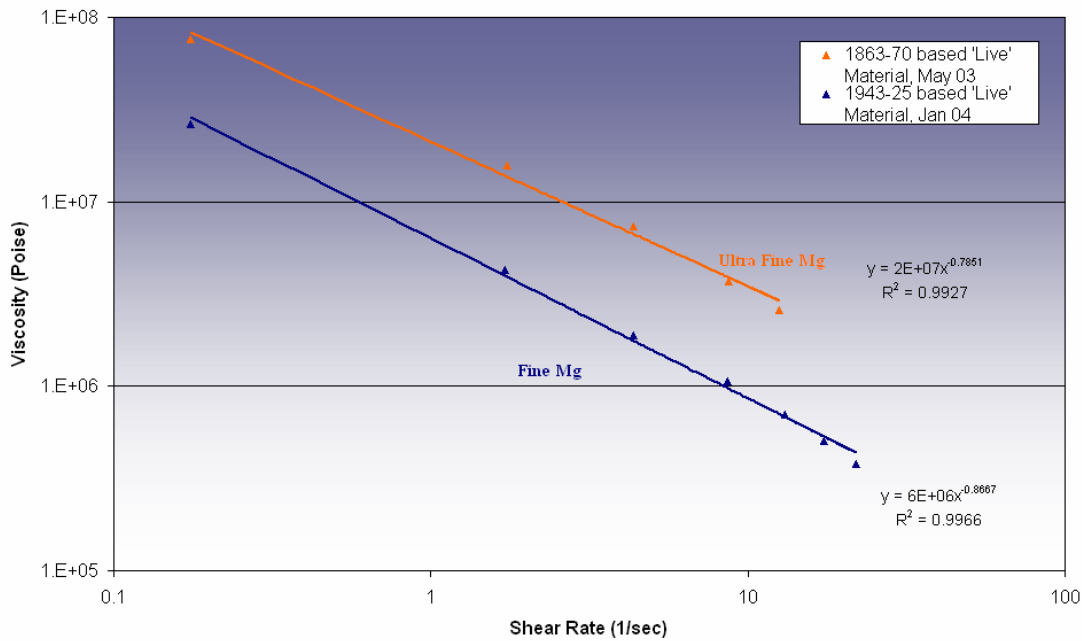


Figure 25. Apparent Viscosity of MTTP Formulations with Different Mg Particle Sizes.

The interesting thing to note with the switch to a larger Mg particle size was the effect it had on the pre-blend. Pre-blend made with the larger Mg experienced polymer segregation in the capillary extruder. This segregation of the polymer lead to a very high apparent viscosity of the pre-blend (see Figure 26). The tendency of the polymer to segregate from the Mg components at these ratios may lead to processing problems in specific processing environments.

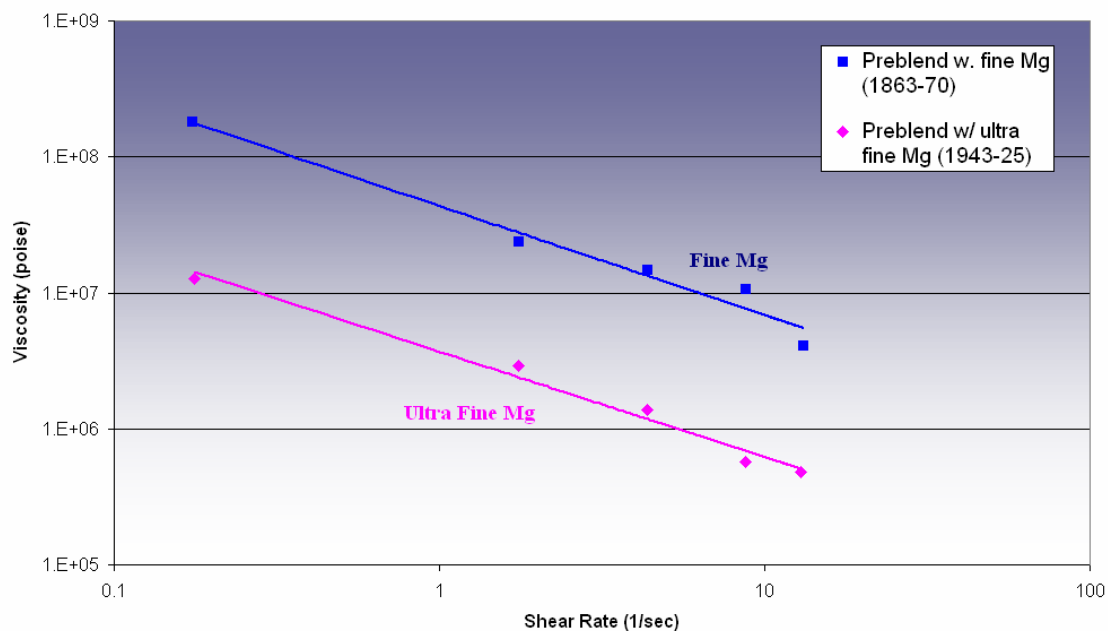


Figure 26. Apparent Viscosity of Pre-blend with Different Mg Particle Sizes.

Following the selection of an adequate pre-blend, inert extrusion evaluations were resumed. Initial inert evaluations with various pre-blend formulations had involved using the standard 19-mm barrel. This barrel has one open feed-port and one open port for a vent port stuffer vacuum line. Because it would not be necessary to pull vacuum on this formulation, the vent port was capped. Evaluations to this point had involved using only one feed stream and for a variety of reasons it was determined that it would be more efficient to use the barrel the containing one feed-port to perform evaluations with the pre-blend. It was not anticipated that the transition to the barrel with two feed ports would present difficulty.

With the change over of the 19-mm TSE upper barrel an inert extrusion was attempted. The use of pre-blend 1943-25 material was held constant and potassium bi-carbonate was substituted for the Teflon[®]/graphite blend. Pre-blend was fed into the rear port and potassium bicarbonate was fed into the second feed port after pre-blend material was feeding from the end of the extruder. After several minutes of run time there was no discernable change in the consistency or color of the material exiting the extruder. Based on past experience feeding materials into the 19-mm TSE it was assumed that potassium bi-carbonate was hanging up in the feed funnel resulting in a limited amount of material making it into the extruder.

Investigation of the feed funnel revealed that material was experiencing difficulty entering the extruder but not as a result of hang-up in the funnel. Pre-blend (1943-25) material had begun creeping into the second feed port before the start of the potassium bi-carbonate feed stream. The creep of this material was so severe that it resulted in the second feed port becoming completely blocked. This blockage of the second feed port and the fill of the screws after the termination of the extrusion can be seen in Figure 27.

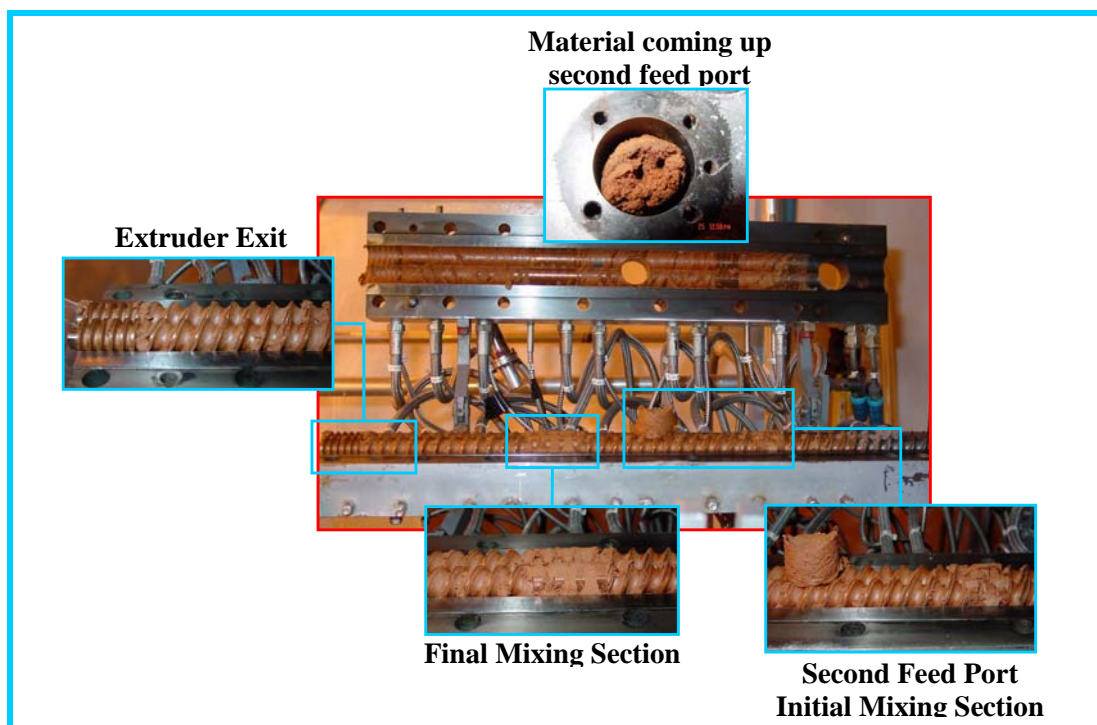


Figure 27. Extruder Following Extrusion of Pre-blend with Two Feed Ports.

When the program was transitioned from the 58-mm TSE to the 19-mm TSE, it was understood that there would be problems due to the limitations of this machine. Very few screw elements are available for purchase off the shelf for the 19-mm. ATK Thiokol Propulsion owns all screw elements available off the shelf, which includes only one pitch of conveying elements. In the 58-mm and other extruders, several pitches of conveying screw elements are available. Typically, under the second feed port, conveying elements with a larger pitch are used to pull the material past this feed port more quickly.

Because only one pitch of these elements was available off the shelf for order, it was necessary to special order elements to meet these needs. High volume screws were designed and manufactured by Material Processing & Research Inc. for use with the 19-mm TSE. It was anticipated that the use of screws such as this could reduce the back pressure seen at the second feed port and therefore reduce the build-up of material.

An initial extrusion attempt was made using the parameters that were established for this process: screw speed of 70-rpm, a temperature profile of 240°F along the entire length of the barrel and a feed rate of 4.05-lbs/hr. These parameters were based on previous experience in the 19-mm TSE and on the experiences with extrusion of the pre-blend with only one feed-port available. The use of these new elements did not eliminate the problem that was experienced. While the use of the new TSE elements did slow the progression of the pre-blend into the second feed-port, it did not prevent the material from eventually building to a point that the feed-port was completely closed off.

Following this extrusion attempt it was apparent that more than a screw design change was necessary to prevent material from crawling into the second feed port. Several processing parameters were evaluated and modified in an attempt to minimize the impact of material being diverted into this port. The first parameter modified was the feed rate of the pre-blend material. The intention was to decrease the amount of material present in the barrels at one time. The slowest feed-rate at which the LIW feeder could feed the pre-blend was determined and established as the feed-rate. This was set at 2.0-lbs/hour.

In a continuing attempt to minimize the amount of material present in the extruder, the extruder speed was altered. The screw speed was increased to the maximum speed that was comfortable for running the extruder. The screw speed was set at 90-rpms.

Continuing optimization centered on the temperature profile of the extruder barrel. The temperatures in the zones were altered in an attempt to determine if changing the temperature could result in a different consistency of material that would be able to pass smoothly under the second feed-port. Table 16 shows the different extrusions that were attempted. None of these alterations was successful in eliminating the creep of the material in the second feed-port. Putting into practice these different parameters was not able to alleviate the problem. The most promising extrusions were seen in the M241-03-006A and H extrusion runs where all zones were set at 240°F. These extrusions were seen as the most promising because the feed-port took longer to plug off than in the others. These parameters were most amicable to preventing the build-up of material in the second feed port and were established as the extrusion parameters for continuing evaluation.

Table 16. Extrusion Temperature Adjustment in an attempt to Minimize to Closing Off of the Second Feed-Port.

Extrusion Number	Zone 1 Temperature	Zone 2 Temperature	Zone 3 Temperature	Zone 4 Temperature	Success?
M241-03-006A	240°F	240°F	240°F	240°F	Promising
M241-03-006B	230°F	230°F	230°F	240°F	No
M241-03-006C	220°F	220°F	230°F	240°F	No
M241-03-006D	210°F	220°F	230°F	240°F	No
M241-03-006E	220°F	220°F	220°F	230°F	No
M241-03-006F	240°F	230°F	220°F	230°F	No
M241-03-006G	200°F	200°F	200°F	200°F	No
M241-03-006H	240°F	240°F	240°F	240°F	Promising

Putting into practice these parameters alone did not alleviate the problem of material creeping up the second feed port; another option needed to be explored. A slanted feed plug was designed and constructed. This feed-plug is shown in Figure 28. This plug covered the top of one complete screw and the corners were slightly rounded. The pre-blend flowed pass the second feed-port with very little difficulty for the majority of this run time. The extruder conditions during the initial extrusion past the slanted feed-port plug is presented in Figure 29. After approximately 25 minutes there began to be some buildup around the feed-port that eventually resulted in the entire feed area becoming plugged. While this extrusion could not be classified as a definite success, it did prove to be very promising.

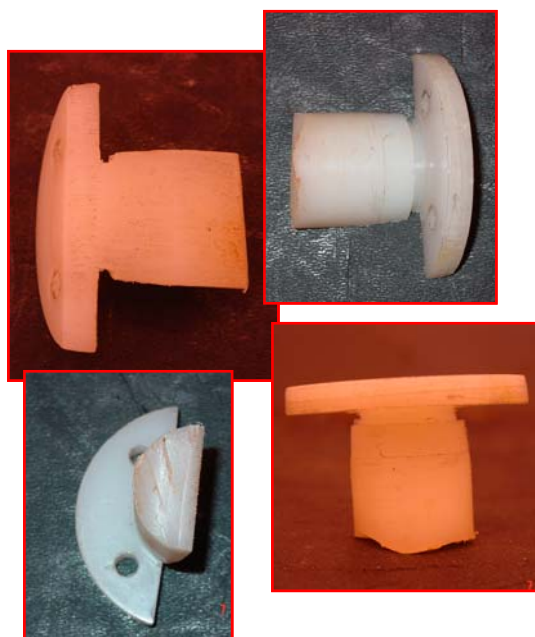


Figure 28. Slanted/Tapered Feed-Plug.

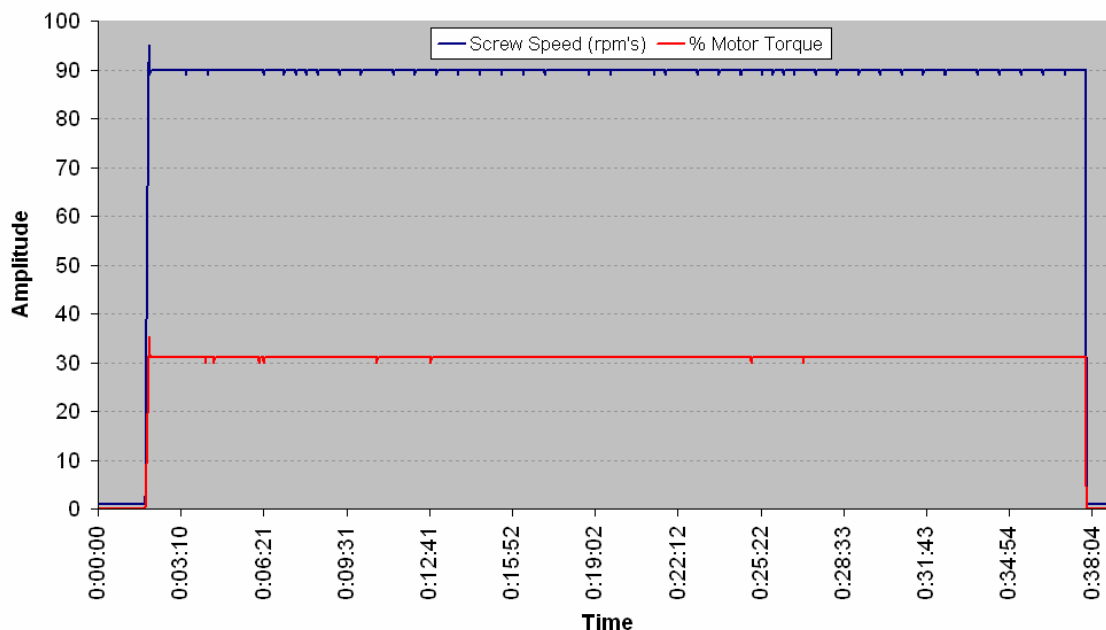


Figure 29. Extruder Conditions during the Initial Extrusion with the Slanted Feed-port Plug.

Following the promising extrusion of the MTTP pre-blend past the slanted feed-plug, inert extrusions were attempted to determine if the feeding of material past this slanted feed-plug would aide in preventing material from creeping up the feed-port and to determine if processing and safety issues would result from the addition of cold, dry materials through the second feed-port. The initial inert extrusion used pre-blend material and potassium bi-carbonate as a substitute for the Teflon[®]/graphite blend. Only limited extrusion was possible with pre-blend and potassium bi-carbonate. Potassium bi-carbonate had difficulty feeding past the slanted feed-plug and extrusions of 5 minutes were the maximum possible before potassium bi-carbonate bridged in the funnel. Figure 30 shows the condition of the extruder during this extrusion. No over-torque conditions were experienced and no obvious changes in the torque were seen as the Teflon[®] substitute was added to the pre-blend.

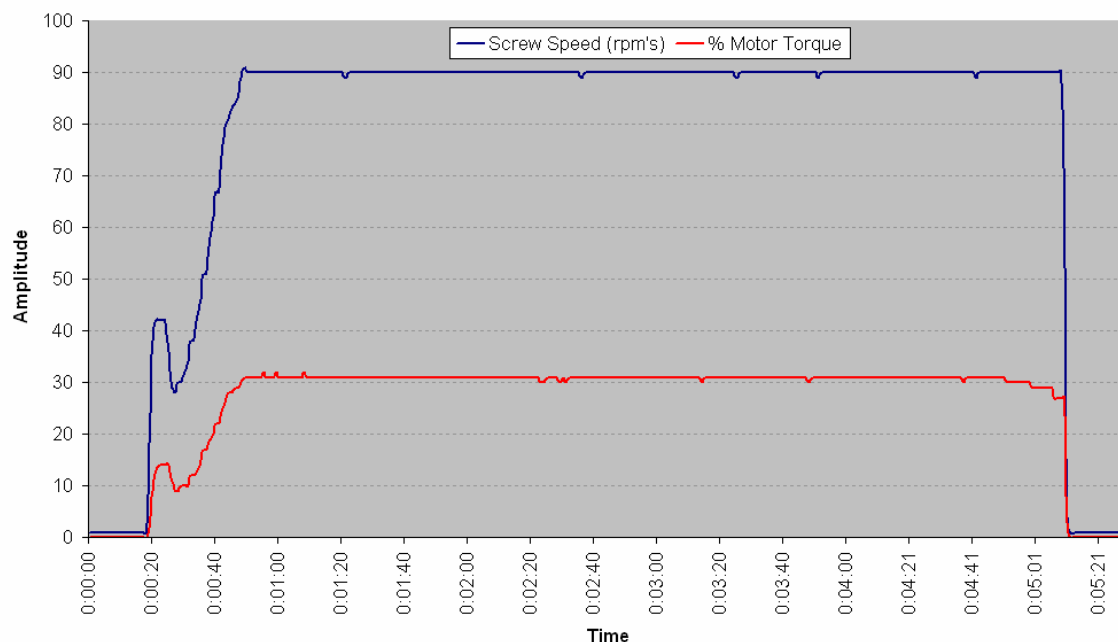


Figure 30. Extrusion of Pre-Blend and Potassium Bi-carbonate. Potassium bi-carbonate is being fed past the slanted feed-plug.

Due in part to the lack of success in feeding potassium bi-carbonate into the extruder, the addition of Teflon[®] to the extruder was evaluated. The initial evaluation with Teflon[®] consisted of feeding Teflon[®]/graphite blend past the slanted feed-plug and into the running extruder. No temperatures were applied to the extruder and down stream mixing elements were removed. Teflon[®]/graphite blend was the only constituent added to the extruder. Teflon[®]/graphite blend was fed into the extruder in this manner without difficulty and no extreme amount of build-up was seen in the feed funnel.

More evaluation of feeding Teflon[®]/graphite blend into the extruder needed to be completed prior to proceeding to extrusion of live material. In order to complete this inert extrusion an inert polymer, OPTEMA TC-220, was substituted for pre-blend. OPTEMA TC-220, or simply TC-220, is an ethylene methyl acrylate copolymer produced by Exxon Mobil Chemical Company. While this material does melt and soften at a much lower temperature than does the pre-blend it was adequate for showing if Teflon[®]/graphite blend could be incorporated past the slanted feed-port plug with a steady stream of material passing underneath. Extrusion data from the run is shown in Figure 31. The extruder was run with TC-220 and Teflon[®] for well over an hour without incident. TC-220 did not climb the second feed-port and the Teflon[®]/graphite blend had no difficulty feeding past the slanted feed-plug. No over-torque conditions were seen during the extrusion run. This inert extrusion provided the necessary confidence for proceeding with a live run.

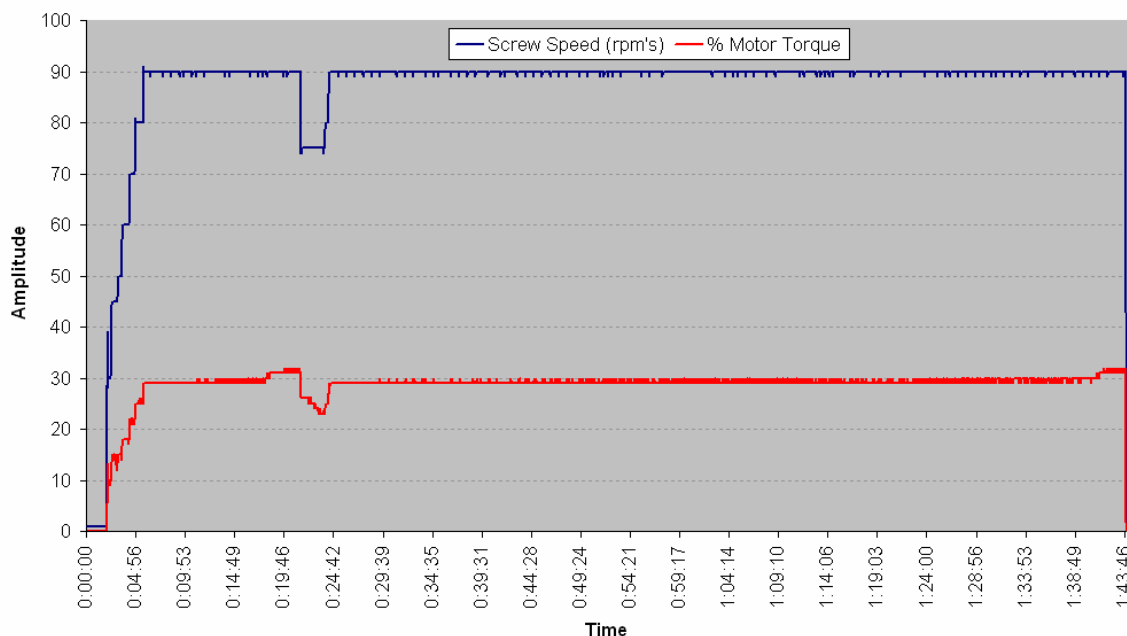


Figure 31. Inert Extrusion: Teflon®/graphite and TC-220 as Polymer Substitute for Pre-blend.

Required Processing Safety Reviews

Following the inert extrusions it was time to take the process live. Several levels of safety review were required at ATK Thiokol prior to beginning the live extrusion. These reviews involved all levels of management.

Initial evaluations centered around peer reviews and verification that all policies and procedure for scaling up energetic materials, as well as for using the 19-mm TSE were being followed. These peer reviews included the MTTP Thiokol team, direct supervision and Research and Development (R&D) safety personnel. Due in part to the bulletin released by Kilgore, the team decided to take the safety evaluation to the next level of scrutiny, the Safety Advisory Committee (SAC).

The R&D-SAC includes members at all levels of management in the ATK Thiokol R&D Lab. The group met in the building that the process was to be performed in to be sure that all possible precautions were being taken with handling this material. The process was approved, conditional upon subsequently completed action items. All action items from the R&D-SAC were completed.

Following the completion of the action items from the R&D-SAC the process was taken to the Plant Process Control Board (PPCB) for approval. The PPCB includes several levels of management and safety personnel from the ATK Thiokol campuses and is required for processes that present unique hazards. Following the PPCB review the extrusion was approved with no action items.

Several small extrusion runs were completed after the approval from the PPCB. Following these initial extrusions, additional management scrutiny was required. For additional extrusion to occur, it was necessary for the process to be approved by the Review Board. This Review Board includes the most upper levels of management, including the Vice President of ATK Thiokol. Upon presentation of the MTTP process to the Review Board, the process was approved for full processing.

Live Extrusion

The basic MTTP process for the 19-mm TSE is a three-step process (Figure 32). While this process is ideal for use with the 19-mm TSE, a more robust machine may be able to process the material without the use of a pre-blend, therefore reducing the processing steps from three to one.

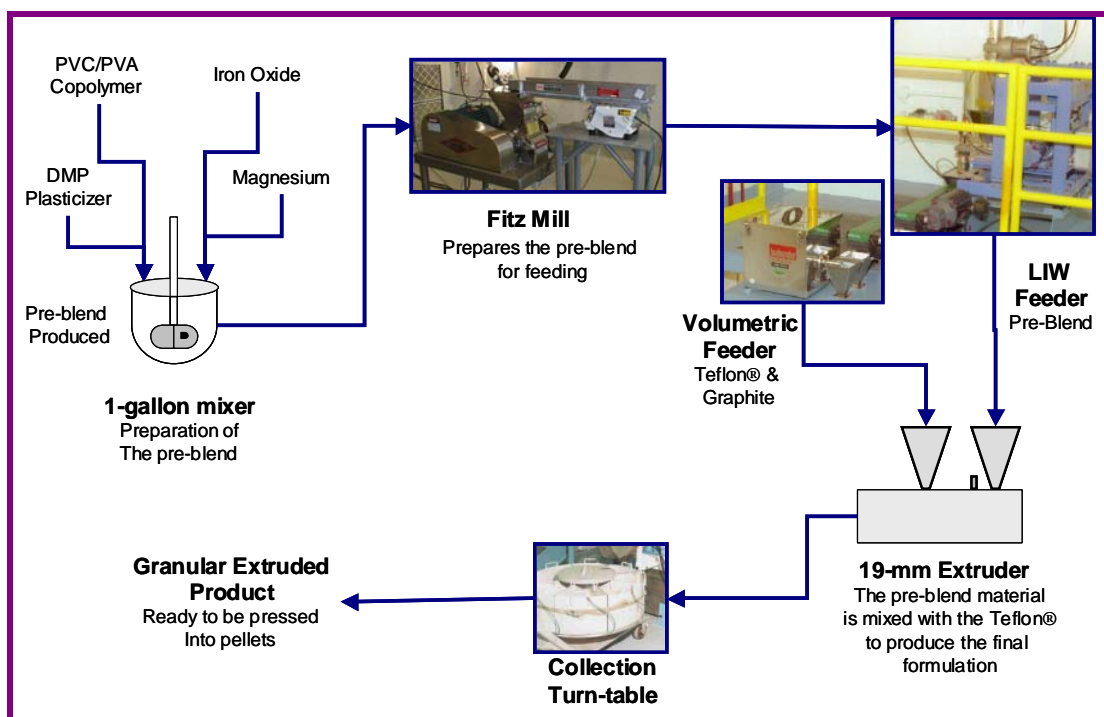


Figure 32. MTTP Process Flow.

Prior to extrusion, the two feed streams need to be prepared. The pre-blend is prepared in a 1-gallon mixer without the use of a solvent. This preparation consolidates the TP, plasticizer, magnesium and iron oxide. The benefit of this consolidation beyond that it allows the material to be processable, is that it reduces the ESD hazards associated with extrusion by coating the magnesium prior to its being fed into the TSE. Once the pre-blend has been produced in the 1-gallon mixer, it is necessary to take this material to the Fitz-mill for sizing. The material removed from the 1-gallon mixer is not of a consistency that makes for easy feeding from the 20-mm LIW. This Fitz-milling results in a consistent feedstock that is ideal for feeding from the 20-mm LIW into the 19-mm TSE. Graphite and Teflon® are also consolidated prior to the extrusion operation. This occurs in a small V-shell blender. Teflon®/graphite feeds very easily from the volumetric feeder and as consistently as can be expected with this piece of equipment.

MTTP pre-blend is fed from the 20-mm LIW feeder to a baffled funnel upstream of the Teflon[®]/graphite addition (Figure 32). A basic solid ingredient feed-port is used to introduce the pre-blend to the extruder. Conversely, the Teflon[®]/graphite is fed into the TSE using a straight or standard funnel. The standard funnel is acceptable for the Teflon[®]/graphite addition because even if a fire should happen to propagate up to the volumetric feeder there is not a threat of the Teflon[®]/graphite alone transitioning to detonation. As is stated above, the feed-port for the Teflon[®]/graphite addition is interesting. A tapered feed port plug that closes off half of the feed port is used. This feed-port plug allows the Teflon[®]/graphite to flow easily into the TSE while preventing the back-flow of pre-blend into the feed-port.

The screw design used in the 19-mm is presented in Figure 33. This configuration contains two mixing sections. The first mixing section follows pre-blend addition and softens this material before Teflon[®]/graphite addition. These are neutral mixing elements. The second mixing section consolidates pre-blend with Teflon[®] and graphite, these are also neutral mixing elements. The newly obtained conveying elements are used to feed pre-blend material more efficiently. This screw configuration limits the length of time that live material is in the extruder.

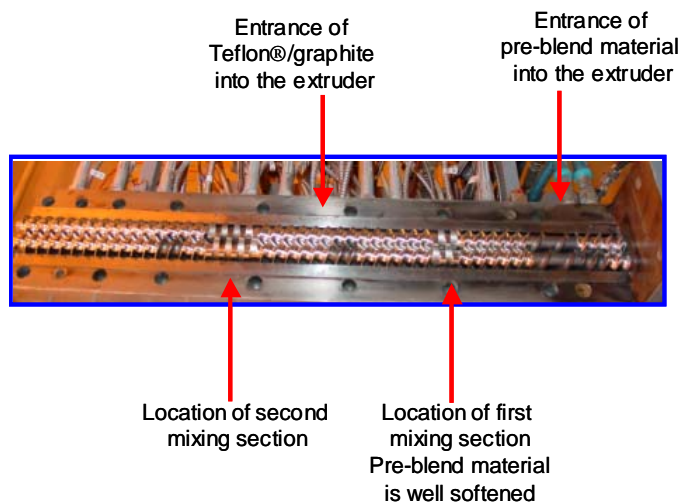


Figure 33. MTTP Screw Design for the 19-mm TSE.

Due the possibility of material climbing into the second feed-port, a system was devised to monitor this port. As is seen in Figure 34, a mirror and lighting system was set up with the remote camera monitoring system to allow for the supervision of this port. From the safety of the control bunker it is possible for operators to observe the progression of material in the TSE. Even when Teflon[®]/graphite blend is being added it is still possible to monitor this port.

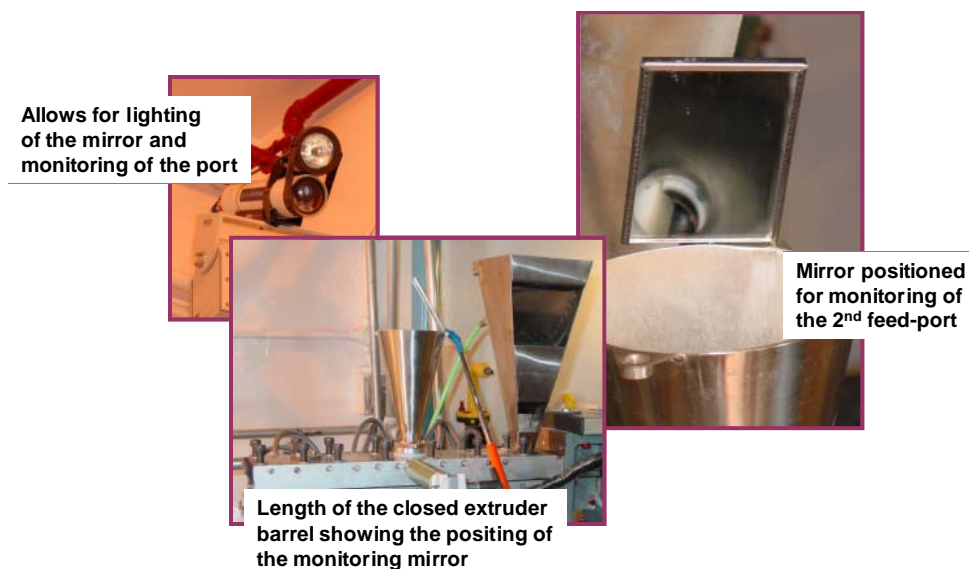


Figure 34. Set-up of Second Feed-Port Monitoring System.

The extrudate collection was a more complex enterprise (Figure 35). Due to the recommendation to limit accumulation to 2-lbs of material or less a collection system needed to be devised that would allow for the isolation of 2-lbs of material while allowing for an uninterrupted extrusion. Material exited the extruder onto a slide that led to a turntable containing eight individual trays. At start-up and shut down, the material is directed into a container that is classified as waste. The waste container will not contain large amounts of full formulation MTTP. The turntable containers have velostat liners to receive and store extrudate and these liners are grounded. After approximately 2-lbs of material is added to a collection container, the turntable is turned to begin extrudate collection in a different container. The sizes of the containers are such that it allows for the collection of upward of 25-lbs of material. Due to the large amount of material that can be collected, there is adequate isolation of 2-lbs collected in the individual trays.

Once adequate material has been extruded to fill the collection trays, the extruder is purged and operators enter the building to package and remove material. For this operation personnel enter the building in full pyro-suits. One collection tray of material is handled at a time. As material is removed from the collection trays, it will be weighed and packaged for shipment. At this time samples for burn-rate testing are taken. Once material is packaged for transport it can be considered safe to handle and the 2-lb limit for safety is no longer mandatory, however, care should be taken when handling the boxes.



Figure 35. Photo of 19-mm TSE Setup for MTTP Extrusion, Including Collection Turntable.

Several live extrusion runs were performed. Initial extrusions were not as efficient as would have been desired. The second feed-port was found to close off much more quickly than had been anticipated. Very rudimentary cleaning of this feed-port did not extend the life of the extrusion run to any great extent. While several pounds of successful extrusion were obtained, lengthy extrusion runs were not possible. It was determined that a through cleaning of the feed-ports and extruder barrel would be more likely to increase the extrusion time.

MTTP Production Results

Due to the limited run times described above, the material for shipment to ARDEC and Crane was produced in a series of short TSE runs. The burn times and radiant intensities were measured for each run, Table 17. There is variability between the runs, likely caused by difficulty in maintaining a consistent feedstream. The data from the TSE runs can be compared to two types of typical MTV compositions. It can be seen that the burn times of the MTTP composition are longer than desired, although the total radiant energy is similar to, or better than, typical MTV. Additional formulation work would be necessary to match the performance exactly, however the MTTP approach does appear to be feasible. In preparation for shipping, the lots were sorted according to performance, Table 18.

Table 17. MTTP TSE Production Data.

Formulation ID	Ave Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)	Performance Factor (Radiant Energy/Ave Burn Time)
71 TSE Lot #2	10.13	146	259	25.6
71 TSE Lot #2b	11.66	135	277	23.8
71 TSE Lot 4	12.24	97	212	17.3
71 TSE Lot 6	11.57	104	215	18.6
71 TSE Lot 2D	13.13	108	248	18.9
71 TSE Lot 2E	13.02	107	241	18.5
71 TSE Lot 2F	9.65	135	229	23.7
71 TSE Lot 4F	9.49	144	241	25.4
71 TSE Lot 5D	11.94	120	250	20.9
71 TSE Lot 5E	10.53	98	184	17.5
71 TSE Log 5F	9.84	138	239	24.3
71 TSE Log 7D	13.30	105	242	18.2
71 TSE Log 7E	8.88	147	230	25.9
71 TSE Lot 8E	10.12	101	182	18.0
71 TSE Lot 4B	11.69	84	176	15.1
71 TSE Lot 4H	12.10	74	162	13.4
71 TSE Lot 2C	10.88	107	210	19.3
71 TSE Lot 2H	10.80	128	245	22.7
Average	11.2	115	225	20
Deviation	1.3	22	31	4
MTV (1C)	4.87	264	212	43.6
MTV (1A)	3.1	315	185	59.0

Table 18. TSE Production Lots Sorted Relative to Performance.

Formulation ID	Ave Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)	Performance Factor (Radiant Energy/Ave Burn Time)
71 TSE Lot 4H	12.10	74	162	13.4
71 TSE Lot 4B	11.69	84	176	15.1
71 TSE Lot 4	12.24	97	212	17.3
71 TSE Lot 5E	10.53	98	184	17.5
71 TSE Lot 8E	10.12	101	182	18.0
71 TSE Lot 7D	13.30	105	242	18.2
71 TSE Lot 2E	13.02	107	241	18.5
71 TSE Lot 6	11.57	104	215	18.6
71 TSE Lot 2D	13.13	108	248	18.9
71 TSE Lot 2C	10.88	107	210	19.3
71 TSE Lot 5D	11.94	120	250	20.9
71 TSE Lot 2H	10.80	128	245	22.7
71 TSE Lot 2F	9.65	135	229	23.7
71 TSE Lot #2b	11.66	135	277	23.8
71 TSE Lot 5F	9.84	138	239	24.3
71 TSE Lot 4F	9.49	144	241	25.4
71 TSE Lot #2	10.13	146	259	25.6
71 TSE Lot 7E	8.88	147	230	25.9

The lots from the TSE runs were sorted for shipment such that the different lots could be blended by ARDEC and Crane to produce blends that were similar in performance, Table 19.

Table 19. Shipping Segregation to Normalize Performance of Materials Sent to Crane and ARDEC.

Send to Crane

Formulation ID	Ave Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)	Performance Factor (Radiant Energy/Ave Burn Time)
71 TSE Lot 4B	11.69	84	176	15.1
71 TSE Lot 5E	10.53	98	184	17.5
71 TSE Lot 7D	13.30	105	242	18.2
71 TSE Lot 6	11.57	104	215	18.6
71 TSE Lot 2C	10.88	107	210	19.3
71 TSE Lot 2H	10.80	128	245	22.7
71 TSE Lot #2b	11.66	135	277	23.8
71 TSE Lot 4F	9.49	144	241	25.4
71 TSE Lot 7E	8.88	147	230	25.9

Send to ARDEC

Formulation ID	Ave Burn Time (sec/in)	Radiant Intensity (W/sr)	Radiant Energy (J/sr-g)	Performance Factor (Radiant Energy/Ave Burn Time)
71 TSE Lot 4H	12.10	74	162	13.4
71 TSE Lot 4	12.24	97	212	17.3
71 TSE Lot 8E	10.12	101	182	18.0
71 TSE Lot 2E	13.02	107	241	18.5
71 TSE Lot 2D	13.13	108	248	18.9
71 TSE Lot 5D	11.94	120	250	20.9
71 TSE Lot 2F	9.65	135	229	23.7
71 TSE Lot 5F	9.84	138	239	24.3
71 TSE Lot #2	10.13	146	259	25.6

The quantities of each lot are shown in Table 20. Slightly over 8 lbs was packaged for ARDEC; slightly over 11 lbs was packaged for Crane. A Interim Hazards Classification, 1.1G, was obtained from the Navy, and the material was shipped to ARDEC and Crane for their evaluation efforts.

Table 20. Lots and Quantities for Shipping to Crane and ARDEC.

Send to Crane	Weight (gms)	Weight (lbs)
71 TSE Lot 4B	12.56	0.03
71 TSE Lot 5E	673.13	1.48
71 TSE Lot 7D	184.54	0.41
71 TSE Lot 6	55.48	0.12
71 TSE Lot 2C	413.5	0.91
71 TSE Lot 2H	556.9	1.23
71 TSE Lot 2B	322.51	0.71
71 TSE Lot 4F	749.78	1.65
71 TSE Lot 7E	754.95	1.66
TOTAL	3723.35	8.21

Send to ARDEC	Weight (gms)	Weight (lbs)
71 TSE Lot 4H	629.38	1.39
71 TSE Lot 4	130.4	0.29
71 TSE Lot 8E	217.77	0.48
71 TSE Lot 2E	708.1	1.56
71 TSE Lot 2D	447.39	0.99
71 TSE Lot 5D	868.63	1.92
71 TSE Lot 2F	789.86	1.74
71 TSE Lot 5F	840.83	1.85
71 TSE Lot 2	495.74	1.09
TOTAL	5128.1	11.31

MTTP Combustion Residue Analyses

A sample of ash from the MTTP combustion process was submitted for analysis in hopes of determining if any dioxins or furans are present.

An attempt was made to essentially follow EPA Method 8280 for polychlorodibenzodioxins (dioxins) and polychlorodibenzofurans (furans). Method 8280 calls for analysis using a high-resolution GC with a high-resolution mass spectral detector. Method 8080, which is used for the analysis of polychlorinated biphenyls (PCBs) calls for analysis using a GC equipped with an electron capture detector (GC/ECD). The ECD is very sensitive to halogenated compounds. We have had experience analyzing transformer oil samples following Method 8080 but limited experience analyzing for dioxins or furans. The initial approach was to prepare the sample following Method 8280 and analyze the sample per Method 8080 using GC/ECD.

Method 8280 references many matrices typical of waste from incinerators: fly ash, still bottoms, air samples, etc. This sample was treated as a “fly ash”. This called for dilution in toluene followed by filtration. In addition to the ash sample, a second aliquot was spiked with 200 uL of a 5.0 µg/ml dioxin and furan mix. The spike consisted of a 5-component dioxin standard and a 5-component furan standard, each component at 1.0 µg added to one gram of sample. The spike

was also diluted in toluene and filtered. A 2-point curve was generated for both dioxin and furan using standard concentrations at 5.0 and 0.5 µg/gram (ppm).

Analysis of the spike sample clearly showed the 5-component dioxin and furan compounds. There does not appear to be any of these compounds present in the sample especially if the sample chromatogram is overlaid with the spike chromatograms. GC/ECD analysis is typically performed by injecting a sample followed by elution through two different phase GC columns. Therefore, target compounds will elute at different times on the two columns providing conformational analysis – if a peak is seen on one column but not the other, then the peak does not pertain to a target compound and is most likely a contaminant. Whenever a peak in the sample eluted at about the same time as one of the target dioxin or furan peaks on one column, it was not found on the second column.

It was concluded through GC/ECD analysis that no dioxins or furans could be detected in the sample. Following the sample preparation method used and estimating against the concentration of target compounds in the spikes, the approximate detection limit is projected to be about 0.1 ug per gram of sample (0.1 ppm). In a clean matrix the detection limit would be much lower.

NSWC Crane: Evaluation of MTTP

Hazards sensitivity and DTA tests were completed on the MTTP composition send to NSWC Crane (Table 20) and on the baseline IR flare composition 757JC for comparison. The sensitivity test results obtained at NSWC Crane are shown in Tables 21-24. The MTTP composition was slightly less sensitive than the baseline IR Comp 757JC to impact, friction and electrostatic testing. The overall rating assigned by NSWC Crane was Very Low to Moderate for MTTP, the same as the baseline.

Table 21. NSWC Crane Impact Sensitivity Tests.

Impact Sensitivity	50% Reaction	
	Height (cm)	Energy (J)
RDX 4RC18-160	35.944	7.05
IR Comp 757JC	161.935	31.74
MTTP Comp	178.401	34.97

Table 22. NSWC Crane Friction Sensitivity Tests.

Friction Sensitivity	Energy (ft-lbs)		Response
	Average	Lowest	
RDX 4RC18-160	1352.56	876.84	1 Out of 10
IR Comp 757JC	1284.37	320.00	8 Out of 10
MTTP Comp	2302.66	266.96	3 Out of 10

Table 23. NSWC Crane Electrostatic Sensitivity Tests.

Electrostatic Sensitivity	Max No-Fire Energy (J)
Sample	
RDX 4RC18-160	0.1800
IR Comp 757JC	1.5125
MTTP Comp	1.8000

Table 24. NSWC Crane Sensitivity Rating.

Sensitivity Rating	Impact Sensitivity	Friction Sensitivity		Electrostatic Sensitivity
Sample		Average Rating	Rating of Lowest Response	
RDX 4RC18-160	Moderate	Very Low	Low	High
IR Comp 757JC	Very Low	Very Low	Moderate	Moderate
MTTP Comp	Very Low	Very Low	Moderate	Moderate

The DTA test results are shown in Figure 36. The differences in the traces are due to the differences in binder decomposition. The MTTP formulation has a small exotherm at ~255 C (490°F), while the baseline 757JC formulation has a very small exotherm at ~350 C (662°F); all other exotherms occur at >500 C (~930°F). The material would be considered acceptable for further testing at Crane.

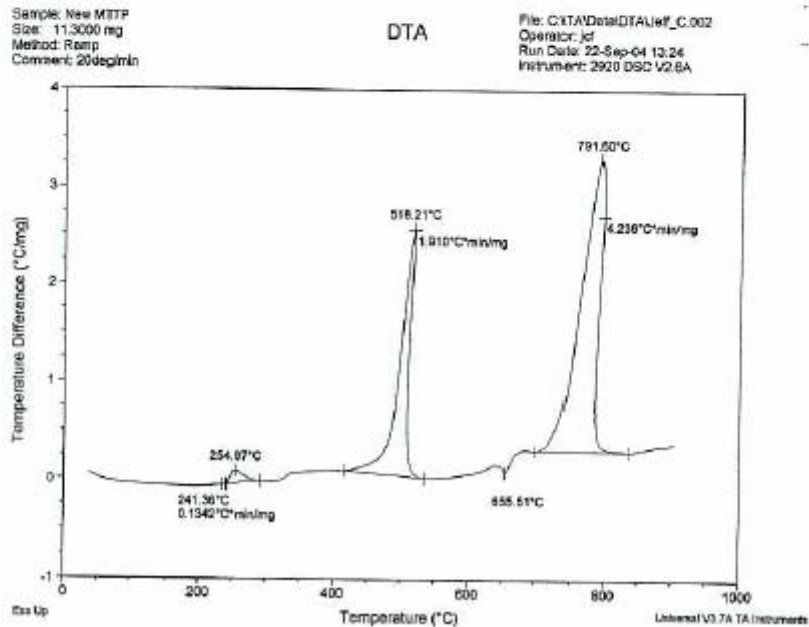
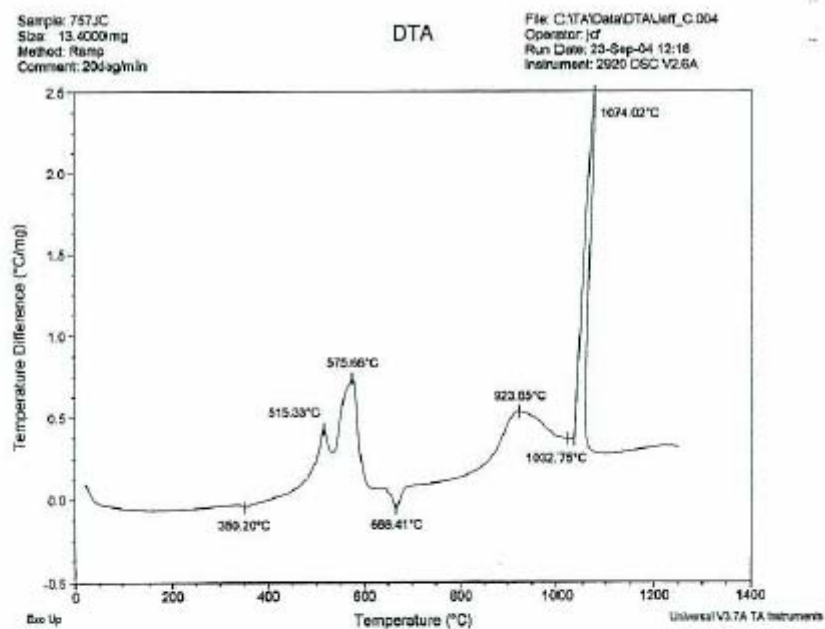


Figure 36. DTA Traces of IR Comp 757JC (top) and MTTP Comp (bottom).

U.S. ARMY RDECOM ARDEC, Picatinny: Evaluation of MTTP

MTTP Test Sample Preparation

The MTTP mixes for full-up flare fabrication and performance testing were produced from an ATK-Thiokol (at Utah) 19mm twin screw extruder. They have a brown color and consist of very small to roughly ¼” size pieces. The MTTP mixes received were in 8 bags each having a different weight of mix along with slight differences in color and physical size of the pieces of the mix. There was also one plastic container that had the capillary extruded mix in a coil (before being broken into pieces). Table 25 below contains the physical descriptions of each bag/container received. The total weight of mix received was roughly 10 pounds. All 8 bags were blended together on rollers for 2 hours before being used for any testing.

Table 25. Physical Description of MTTP Samples.

Bag Label	Weight of Mix (g)	Color	Other Info
MTTP #4	30.661	light brown	small pieces, shavings
MTTP #4	70.268	light brown	capillary extruded (spiral strand of mix)
MTTP #2F	789.06	brown	larger pieces/chunks
MTTP #4H	617.39	red/brown	medium size pieces
MTTP #2D	446.835	brown	larger pieces/chunks
MTTP #5F	843.83	red/brown	medium and large pieces
MTTP #2E	706.24	red/brown	larger pieces
MTTP #8E	216.66	red/brown	larger pieces
MTTP #5D	880.87	brown	larger pieces

Determination of MTTP Flare Loading Pressure

Crush testing was performed first on this mix to establish the loading requirement for full size flare. Ten cylindrical crush test pellets were made, each pellet having a height and diameter of 0.5 inches. Five of these pellets were consolidated at 12000 psi (the nominal MTH flare consolidation pressure) while the last five were consolidated at 15000 psi. This was done to investigate if there was any difference in the strength of the pellets. The weight of each pellet was 3 grams. Five pellets were also made from a US Army standard MTH mix prepared at Picatinny Arsenal. The MTH pellets were consolidated at 12000 psi and tested to establish a baseline.

The actual testing was performed on an Instron Universal Testing Machine, as shown in Figure 37. A pellet was placed on the bottom plate while the top plate with a load cell was positioned manually to be just above the pellet. On the computer used to operate the Instron, the start button was activated and the top plate moved in a slow downward motion at a rate of 0.125 inch per



Figure 37. Instron Universal Testing Machine.

second. When the load cell exerted a force on the pellet it cracked on the sides and flattened. The force and displacement data were recorded on the computer, and a graph was also generated. Figure 38 below shows a graph of pellet number 1 from the 12000 psi consolidation group of MTTP pellets.

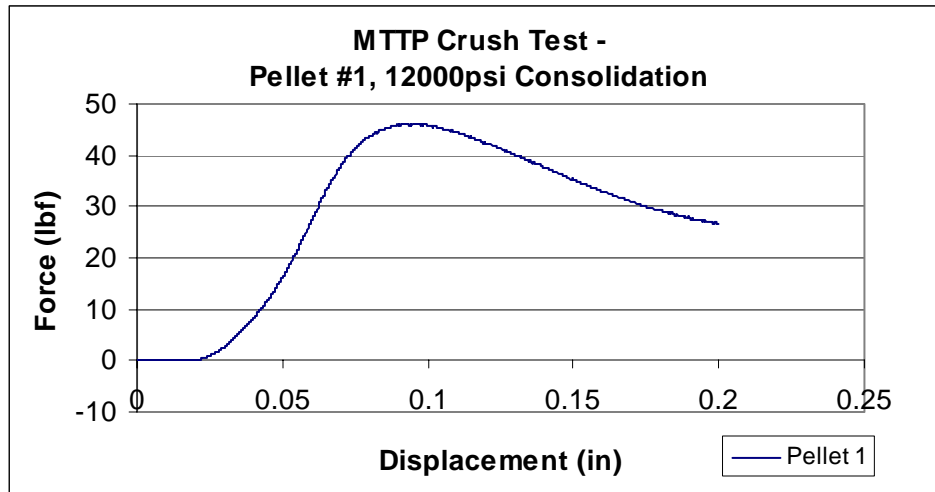


Figure 38. Representative MTTP Crush Test Graph.

Data collected for each pellet tested was the consolidation pressure in psi, weight and height in grams, outer diameter of pellet in inches, and the crush force in pounds force. The density of each pellet was calculated using the weight, height, and outer diameter measurements. The data were summarized in Table 26 below. The crush force recorded was the peak height shown in Figure 38 above.

Table 26. MTTP Pellet Crush Strength Summary.

Mix	Pressure (psi)	Pellet #	Weight (g)	Height (in)	OD (in)	Density (g/cm ³)	Crush Force (lbf)
MTH STD Baseline	12000	1	3.003	0.5220	0.5010	1.78	227.100
		2	3.009	0.5140	0.5010	1.81	227.100
		3	3.006	0.5240	0.5010	1.78	225.600
		4	3.001	0.5205	0.5010	1.78	225.000
		5	3.002	0.5220	0.5010	1.78	223.100
		AVG	3.004	0.5205	0.5010	1.787	225.580
MTTP	12000	1	3.000	0.5585	0.4985	1.68	46.280
		2	3.006	0.5525	0.4995	1.69	52.320
		3	3.009	0.5545	0.4990	1.69	50.390
		4	3.002	0.5515	0.5000	1.69	54.660
		5	3.005	0.5510	0.5000	1.69	54.390
		AVG	3.004	0.5536	0.4994	1.691	51.608
MTTP	15000	1	3.005	0.5645	0.5000	1.65	44.590
		2	3.009	0.5600	0.5000	1.67	52.620
		3	3.004	0.5510	0.5000	1.69	58.230
		4	3.007	0.5580	0.4980	1.69	47.380
		5	3.005	0.5540	0.4990	1.69	53.960
		AVG	3.006	0.5575	0.4994	1.680	51.356

Determination of MTTP Flare Charge Weight

When crush testing was complete, three full size pellets were pressed in a US Army standard MTH die (one V-shape groove on each side). This was done to find a proper weight of mix for a full size pellet and have the pellet dimensions fall within specifications of the standard MTH flare. The specified cross section dimension is 0.85" max on each side within one hour of pressing, with a target of 0.83-0.1". The first pellet pressed used 95.013g of mix and had a side dimension of 0.776 inches. The second pellet was 102.508g and its dimension was 0.8150 inches. Finally, the third pellet was 110.010g and had a dimension of 0.8370", which fell within the specifications. The length of each pellet was 6.83" that was controlled by the die configuration to meet the specification of 6.847-0.3".

These three pellets were then coated with a US Army standard MTH intermediate charge (IC) and FF, wrapped in a one piece pre-cut aluminum tape, and glued into testing bracket holders. They were used in a trial test at the ARDEC Pyrotechnic Flare Tunnel. The pellets were burned successfully but no data was taken.

Fabrication of Full-Up MTTP Flare

After successful testing the three pellets, 34 full size pellets were fabricated using 110 grams and a dead load of 35 tons (equivalent to 12,000 psi) with a 10 secs dwell time. The pellets were coated with the intermediate charge (Magnesium/Teflon/Viton) and then with the FF on top of intermediate charge (Boron/Magnesium/Potassium Perchlorate/Barium Chromate/Viton A). Weights were recorded after each layer of coating was applied and dried. After coating the pellets were taped with aluminum tapes and glued into testing brackets. Figures 39 through 42 demonstrate the photographs of the coating and taping process.

MTTP pellets 1 through 26 were separated in 3 groups for condition: ambient, -65°F and 135°F. The hot and cold pellets were subjected to 4 hours of conditioning prior to testing in the flare tunnel. Static testing was done starting with number 1 at ambient temperature and alternating the testing temperature every three pellets. Data were not available for pellet 27. Pellets 28 through 32 were tested in the wind stream facility at ambient temperature, while pellets 33 and 34 were used for warmers in flare tunnel testing. In addition, the US Army standard MTH flares were tested to establish the nominal values for performance comparison, five each for static and dynamic test configurations.



Figure 39. MTTP Pellet Coated with both Intermediate Composition and First Fire. Immediate charge can be seen on sides of groove as light gray.



Figure 40. MTTP Pellet End Groove Coated with First Fire Connecting all Four Side Grooves.



Figure 41. Wrapping of Aluminum Tape on MTTP Pellet. Space is left on the bottom for the testing bracket.



Figure 42. MTTP Pellet Coated with Intermediate Charge and First Fire, Taped with Aluminum Tape, and Glued into Testing Bracket.

Static Burn Test

The static burn test was performed in the Pyrotechnic Flare Tunnel (B1515) at Picatinny Arsenal. For this test, the IR and silicon (visible) detectors were placed at a distance of 50 ft from the MTTP pellet that was mounted on a test stand vertically with its axial centerline perpendicular to the detectors. The detectors were then connected to trans-impedance amplifiers via BNC (coaxial) cables. The trans-impedance amplifiers were connected to a redundant data collection system consisting of an oscilloscope and Labview board for further analysis. The pellet was set off using an electric match that was aimed at the item's ignition composition. Test setup is illustrated in Figure 43.

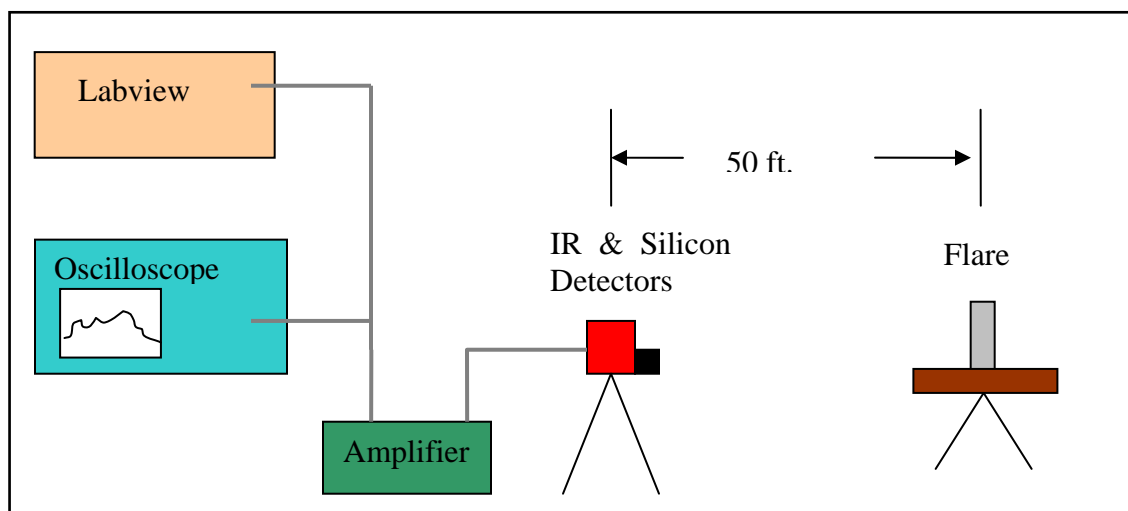


Figure 43. MTTP Flare Static Burn Test Setup.

Dynamic Burn Test

The dynamic test was performed at the Pyrotechnic Wind Stream Facility (B247) at Picatinny Arsenal. For this test, a proper wind speed profile was used to simulate the trajectory of a flare from a rotary wing aircraft's dispenser. The flare was fixed on a test stand in front of the wind tunnel opening. It was ignited via electric match at a predetermined wind speed. The IR and silicon detectors were placed at a distance of 50 ft from the flare mounted on a test stand horizontally. The detectors were then connected to trans-impedance amplifiers via BNC (coaxial) cables. The trans-impedance amplifiers were connected to a redundant data collection system consisting of an oscilloscope and Labview board for further analysis. Test setup is illustrated in Figure 44. All radiometers were calibrated against a blackbody at a known temperature and distance. The silicon detector was calibrated against a NIST traceable 1000 W lamp.

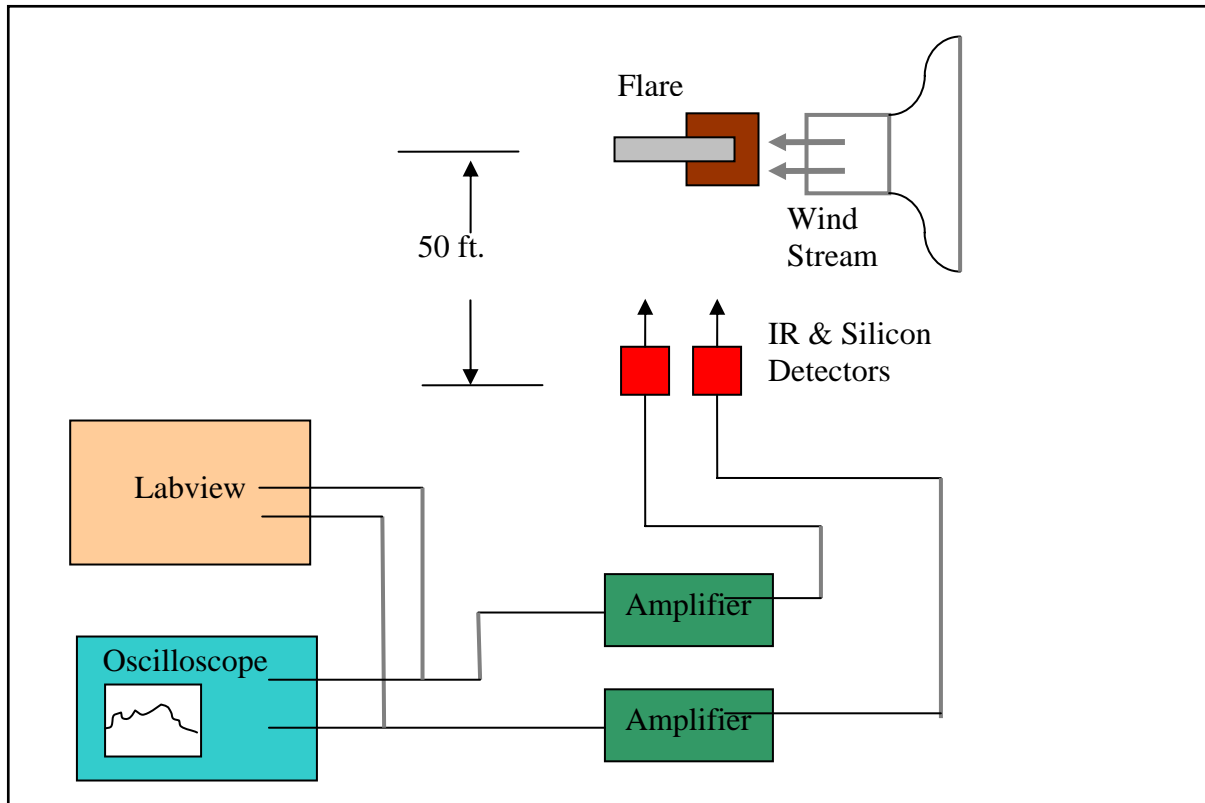


Figure 44. MTTP Wind Stream Dynamic Burn Test Setup.

Burn Test Data Summary

The static and dynamic burn test results were recorded in a form of IR (watts/steradian) versus time (sec) charts. The visible light intensity (candles) versus time data were only recorded for the static test. The integrated IR ((watts/steradian)*sec) and visible light (candle*sec) outputs and burn times were then computed from a programmed Labview software. Each MTTP flare's performance except the burn time was reported as a percent of the nominal value of the standard US Army MTH flare, as shown in the Tables 27 and 28. The typical IR intensity versus time traces for MTTP and standard MTH flares are illustrated in Figure 45.

Table 27. Static Burn Test Data Summary.

Test Sample	% of Nominal Integrated IR Output	% of Nominal Integrated Visible Output	Burn Time (sec)	Test Temp
MTTP_Flare__1.txt	1.10	0.91	3.58	amb
MTTP_Flare__10.txt	1.10	0.87	3.49	amb
MTTP_Flare__11.txt	1.15	0.91	3.60	amb
MTTP_Flare__12.txt	1.13	0.92	3.52	amb
MTTP_Flare__19.txt	1.08	0.86	3.74	amb
MTTP_Flare__2.txt	1.14	1.07	3.54	amb
MTTP_Flare__20.txt	1.10	0.83	3.70	amb
MTTP_Flare__21.txt	1.08	0.83	3.33	amb
MTTP_Flare__3.txt	1.11	0.93	3.34	amb
MTTP_Flare__16.txt	1.19	0.95	3.43	135
MTTP_Flare__17.txt	1.15	1.01	3.49	135
MTTP_Flare__18.txt	1.16	0.94	3.44	135
MTTP_Flare__25.txt	1.14	0.94	3.49	135
MTTP_Flare__26.txt	1.17	1.05	3.56	135
MTTP_Flare__7.txt	1.15	0.90	3.76	135
MTTP_Flare__8.txt	1.16	0.99	3.84	135
MTTP_Flare__9.txt	1.19	1.02	3.80	135
MTTP_Flare__13.txt	1.20	0.99	3.38	-65
MTTP_Flare__14.txt	1.12	0.95	3.28	-65
MTTP_Flare__15.txt	1.03	0.78	3.51	-65
MTTP_Flare__22.txt	1.18	0.93	3.76	-65
MTTP_Flare__23.txt	1.08	0.80	3.99	-65
MTTP_Flare__24.txt	1.08	0.89	3.97	-65
MTTP_Flare__4.txt	1.19	1.02	3.42	-65
MTTP_Flare__5.txt	1.10	1.02	3.35	-65
MTTP_Flare__6.txt	1.07	0.77	3.38	-65
Average	1.13	0.93	3.56	

Table 28. Dynamic Burn Test Data Summary.

Test Sample	% of Nominal Integrated IR Output	% of Nominal Integrated Visible Output	Burn Time (sec)	Test Temp
71205_MTTP_28_5.txt	1.06	N/A	3.44	amb
71205_MTTP_29_4.txt	1.06	N/A	3.39	amb
71205_MTTP_30_2.txt	1.05	N/A	3.65	amb
71205_MTTP_31_3.txt	1.05	N/A	3.29	amb
71205_MTTP_32_1.txt	1.05	N/A	3.27	amb
Average	1.05	N/A	3.41	amb

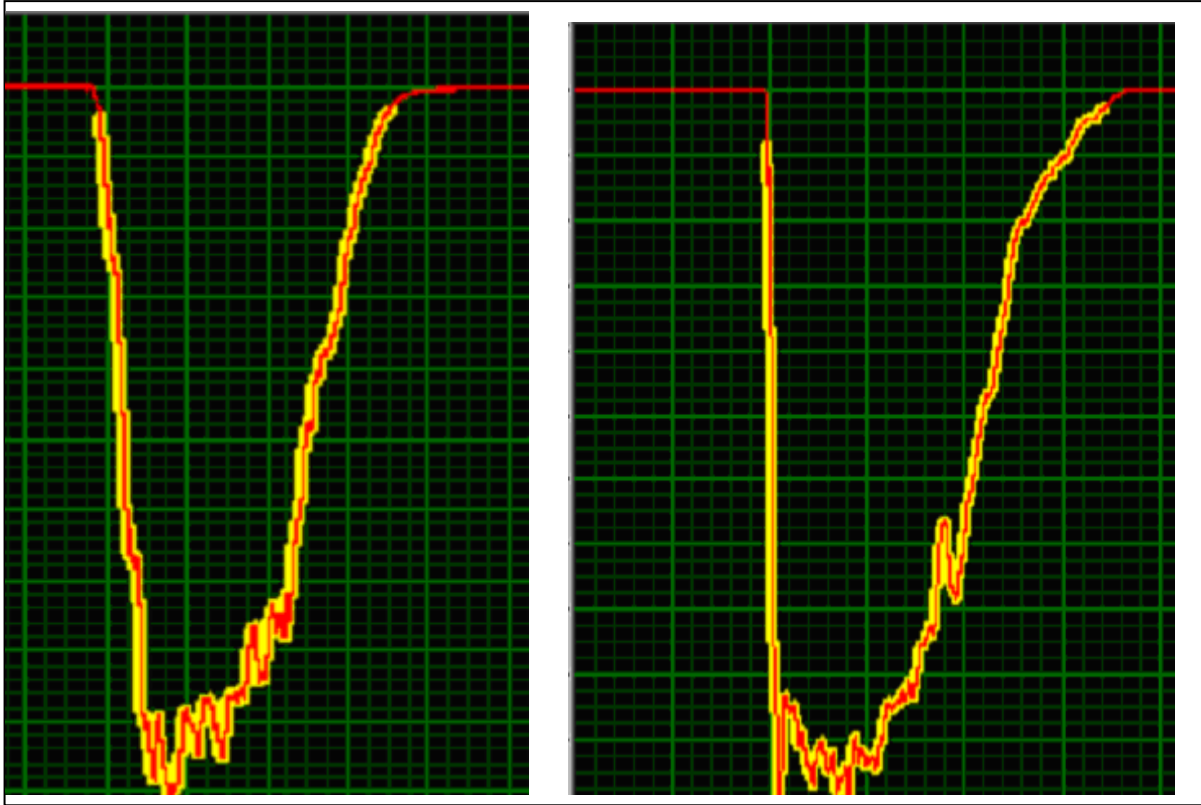


Figure 45. Typical IR Intensity versus Time Traces for MTTP (left, No. 11) and Standard MTH (right, No. 5) Flares.

2.0 SUMMARY AND CONCLUSIONS

An MTTP formulation was developed and processed on a 19-mm TSE. The formulation that processed the best was the PVC-co-PVAc binder system, and it was selected as the formulation of choice. While difficulties were encountered in the extrusion of material, at least in part due to the limitations of the 19-mm TSE, live runs did produce material for shipment to ARDEC and NSW Crane. Combustion residue analysis of the MTTP composition did not show the presence of dioxins or furans.

NSWC Crane evaluation of the material showed the hazards sensitivity and the thermal behavior of the composition were similar to, or slightly better than the 757JC baseline IR composition.

ARDEC evaluated the composition for loading into full-scale articles. The full size MTTP flare consolidation pressure and charge weight were studied to establish the optimal requirements for pellet loading and assembly: 12,000 psi (or 35 dead load) and 110 grams.

The material strength test results indicate that the MTTP flare is significantly lower in crush strength (force) than the standard US Army MTH flare. The use of TP co-polymer with a plasticizer is the most likely contribution factor. It is recommended a transportation vibration test be conducted on the full size MTTP pellets in future efforts such as Pollution Prevention (P2) program, Environmental Security Technology Certification Program (ESTCP), etc.

Thirty-two (32) full size MTTP flares were fabricated with the established loading requirements and the US Army standard MTH flare intermediate charge and FF. Twenty-six (26) were tested for static performance at hot, cold and ambient temperatures and five (5) were tested for dynamic performance at ambient temperature. The IR radiometric and visible light intensity versus time traces were collected and further computed to generate integrated output and burn time data. Results show that the average MTTP IR output slightly outperformed the standard MTH flare under static and dynamic test environments with a respective margin of 13% and 5% while the average burn times for both flares fall within a range of 3.2 to 3.6 seconds. The average MTTP visible light output is 93% of MTH flare. In summary, satisfactory performance was obtained for MTTP flare in both test configurations.